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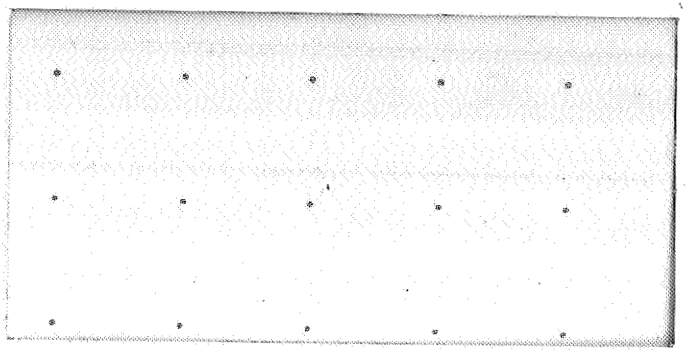

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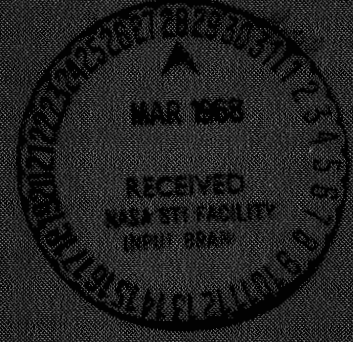

Lockheed

MISSILES & SPACE COMPANY

A GROUP DIVISION OF LOCKHEED AIRCRAFT CORPORATION

SUNNYVALE, CALIFORNIA

21355

2 JULY 1965

LMSC/A758461

BERYLLIUM FABRICATION METHODS DEVELOPMENT PROGRAM

THERMAL TREATMENT

Contract NAS 8-11798

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LOCKHEED MISSILES & SPACE COMPANY
A Group Division of Lockheed Aircraft Corporation
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FOREWORD

This report, submitted under NASA Contract NAS 8-11798, covers Task 2.3, Thermal Treatment, of the Beryllium Fabrication Methods Development Program Plan. The work described herein was performed under the direction of Large Space Vehicles Programs, Space Systems Division, Lockheed Missiles and Space Company. The program encompasses the development and documentation of needed new manufacturing techniques and fabrication methods suitable for the application of beryllium and beryllium alloys in space flight vehicle structures.

The work being accomplished in accordance with the requirements outlined in the Forming (Task 2.1), Metal Removal (Task 2.2), Surface Treatment (Task 2.4), and Joining (Task 2.5) sections of the Program Plan will be reported as each task is completed.

ABSTRACT

This report documents the thermal treatment techniques investigated and evaluated for possible application in the fabrication of beryllium aerospace vehicle structures. The effects of time and temperature on the removal of residual stresses, induced by prior work, are evaluated. The physical, mechanical, and dimensional changes resulting from varying rates of rapid cooling are reported; and the effects of prolonged exposure at high temperatures on the mechanical properties of beryllium sheet are investigated.

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SECTION 1
INTRODUCTION

The objectives of this task are the investigation, evaluation, and correlation of the effects of several types of thermal treatment on the physical, mechanical, and dimensional characteristics of beryllium sheet material, as outlined in the "Thermal Treatment" section of the Beryllium Fabrication Methods Development Program Plan.

The effects of time and temperature on the characteristics of the material were investigated and evaluated. Considerable new work was conducted, on a laboratory scale, to obtain finite data to augment the limited available information. The accomplishment of this task was divided into three well-defined areas, which are reported separately as follows:

Stress Relief	-	determine the time/temperature parameters for the removal of residual stresses induced by prior work.
Cooling Rate	-	determine the effect of various quenching rates/methods on the dimensional configuration and mechanical properties.
Annealing	-	determine the effect of prolonged exposure at elevated temperature in normal atmosphere on the mechanical properties.

SECTION 2
STRESS RELIEF

Beryllium sheet material which has been formed at elevated temperature, but below the recrystallization temperature, will contain residual stresses after it has cooled to room temperature. The elimination of these stresses, in order to realize the optimum mechanical properties of the material, is highly desirable.

2.1 INTRODUCTION

The object of this phase of the program was the determination of the time/temperature parameters required for the relief of the residual stresses induced in beryllium sheet material during hot forming operations.

Two widely used, but entirely different experimental methods were considered for the detection of the presence of residual stresses; i.e., X-ray diffraction, and electrical resistivity. Residual stresses are detected in the X-ray diffraction technique by changes in the lattice parameter, or by changes in the width of the diffraction peak for a particular set of lattice planes. The results of preliminary experiments revealed that the X-ray diffraction method lacked sufficient sensitivity to detect the small changes in the lattice parameter or line width resulting from the stress relieving of the "deformed" specimens. The primary difficulty is that the major changes in the lattice structure occur near the surface of the material; and, because beryllium is very transparent to X-rays, the entire thickness of the specimens is penetrated by the X-ray beams. The separation of the surface effects from the overall or bulk effects is, therefore, extremely difficult.

2.1 INTRODUCTION (Cont.)

The electrical resistivity effect, however, is based upon the change in electrical resistivity which occurs in the "deformed" area as the material is shaped. An "as formed" area has a higher resistance to the flow of an electrical current than an undisturbed area, or material which has been stress relieved. Thus, if the electrical resistance of the same specimens is measured "after forming" and again after "stress relieving," the decrease in electrical resistance is indicative of the decrease in residual stress. The results of preliminary experiments verified that changes in electrical resistance could be detected and measured even though the principal stress effects occur at the surface, and the resistance is measured for the entire specimen. Since the changes in resistance could be correlated with the changes in stress, the "electrical resistivity" technique was employed for this investigation.

Table I presents the certified chemical analyses and mechanical properties of the two lots of material procured for this investigation from the Brush Beryllium Company in accordance with the requirements of Lockheed Specification LAC 07-4008.

2.2 EXPERIMENTAL PROCEDURE

The .50" x 3.00" specimens were cut, using the abrasive wheel technique, from the material and formed into Type "A" and Type "B" specimens at nominal forming temperatures of 1350°F and 1100°F respectively. The Type "A" and "B" specimens are illustrated in Figure 1.

The specimen blanks were cut from the sheet material in the transverse direction, as determined from the dimensions of the original sheet, so that the bends were in the longitudinal direction of the material.

TABLE I

CHEMICAL ANALYSES AND MECHANICAL PROPERTIES - STRESS RELIEF SPECIMENS

VENDOR DATA

Material Identification

Lot No.	1153	9967
Sheet No.	468A	447B
Gauge	.060	.060

Chemical Analyses - %

Be Assay	98.29	98.29
Be O	1.78	1.66
Fe	.124	.138
Si	.040	.050
Al	.055	.095
Mg	.008	.011
Mn	.012	.010
Ni	.023	.021
Cr	.012	.012
C	.111	.120

Mechanical Properties

Sheet No.	Test Direction	Ftu Psi	Fty Psi	Elong. % in 1"
468A	L	75,200	58,300	10.5
468A	T	76,400	57,400	11.0
447B	L	78,300	57,400	11.5
447B	T	86,200	58,100	14.0

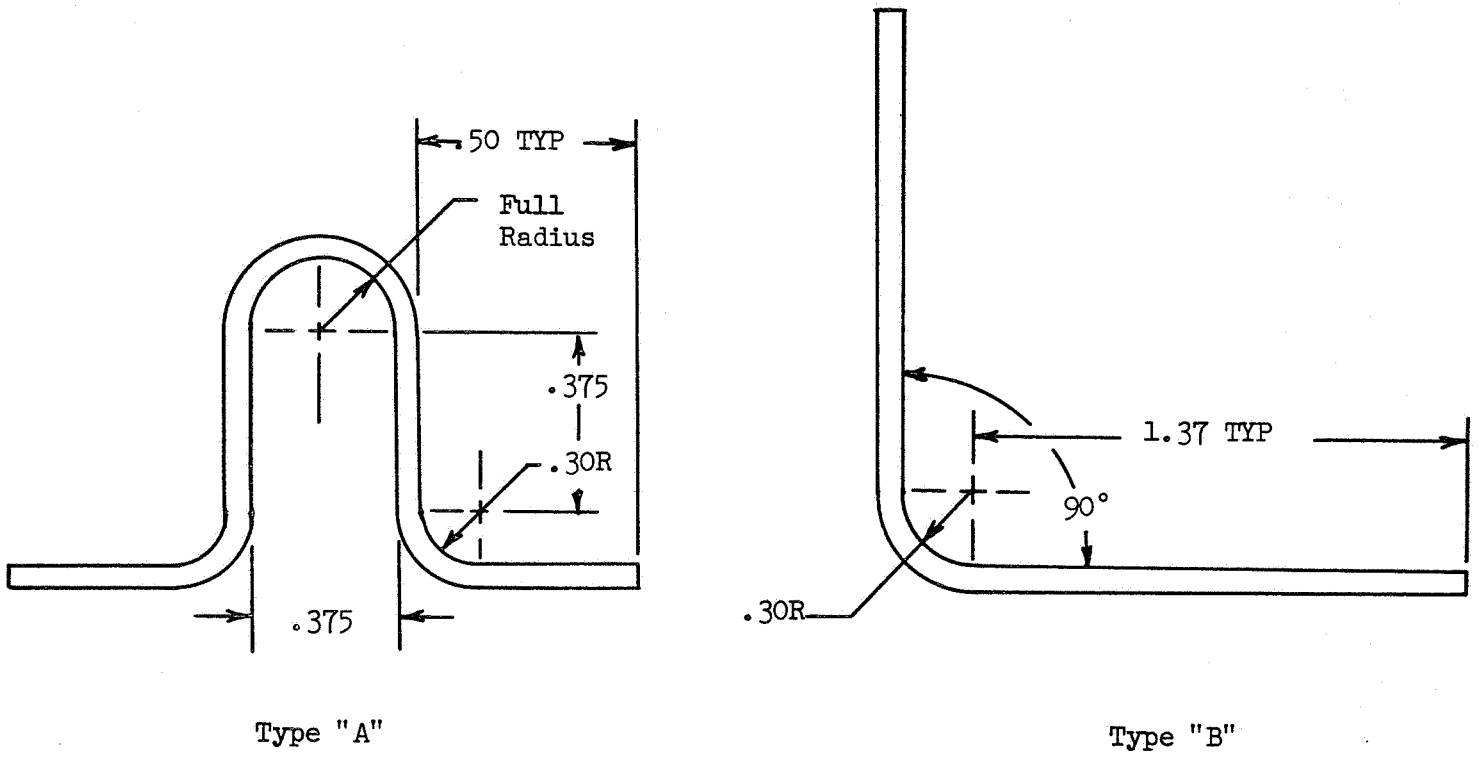
2.2 EXPERIMENTAL PROCEDURE (Cont.)

The layout of the specimens on the sheet material is illustrated in Figure 2.

The individual specimens were formed on a universal type punch and die set consisting of heated "glass rock" die segments and a conduction heated steel punch mounted between the upper segments. The specimen blanks were placed in position on the die, heated by conduction from the hot die, and formed as soon as the desired material temperature had been attained. During the forming operation, the temperature was monitored by means of Type "K" chromel-alumel thermocouples attached to control specimens and recorded on a "Wilco" 12-channel, 0-2250°F range, strip recorder. The universal forming die is illustrated in Figure 3.

Following the completion of the forming operations, the electrical resistance of each specimen was measured and recorded. Since good electrical connections are mandatory for this type of measurement, the contact areas of the specimens were cleaned by etching in a solution consisting of 40 percent nitric acid (HNO_3), 3 percent hydrofluoric acid (HF), and 57 percent water. The "formed" areas were not etched as this would have resulted in the removal of that portion of the material containing the highest level of residual stress.

Figures 4 and 5 illustrate the two types of specimens mounted in the knife-edge test fixtures specifically designed, and fabricated, to insure that the voltage drop was measured over a constant length for each specimen. A Kelvin bridge and associated equipment, illustrated in Figure 6, were used to determine the resistance of each specimen.



Type "A"

Type "B"

Figure 1. Stress Relief Specimens

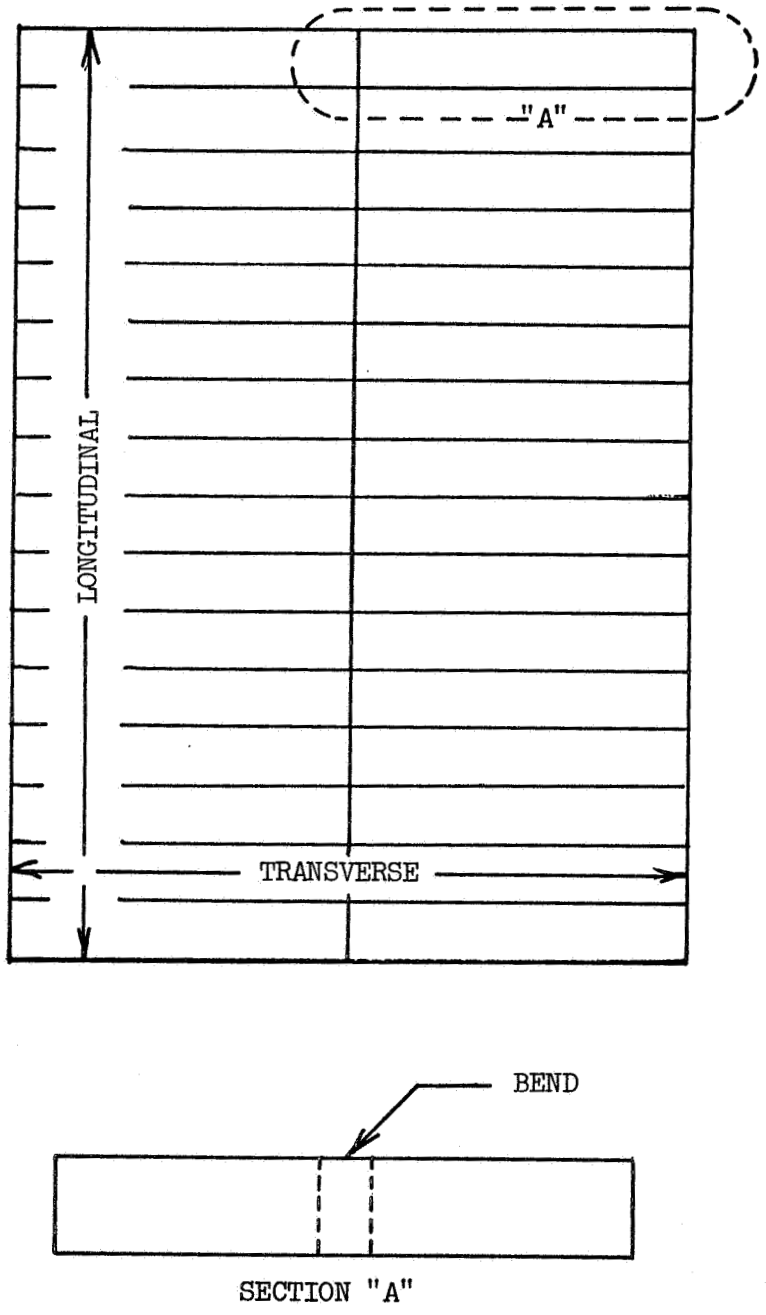


Figure 2. Stress Relief Specimen Orientation

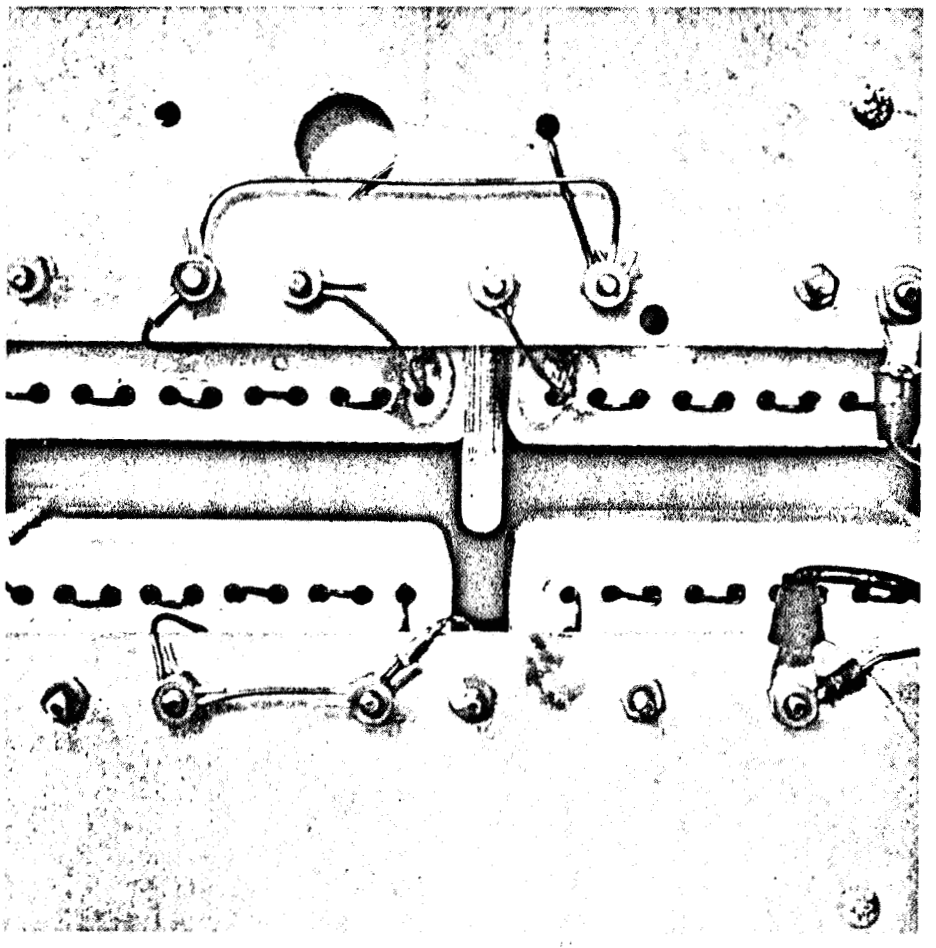


Figure 3. Universal Forming Die

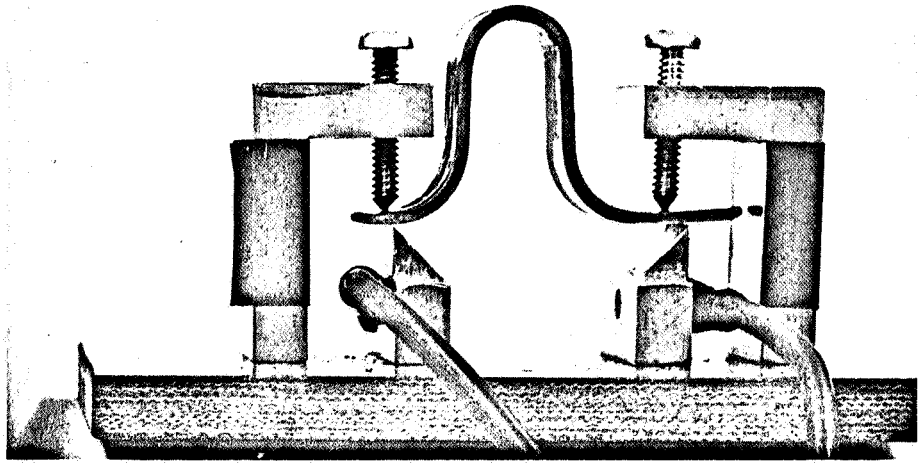


Figure 4. Type A Specimen Mounted in Knife-edge Test Fixture

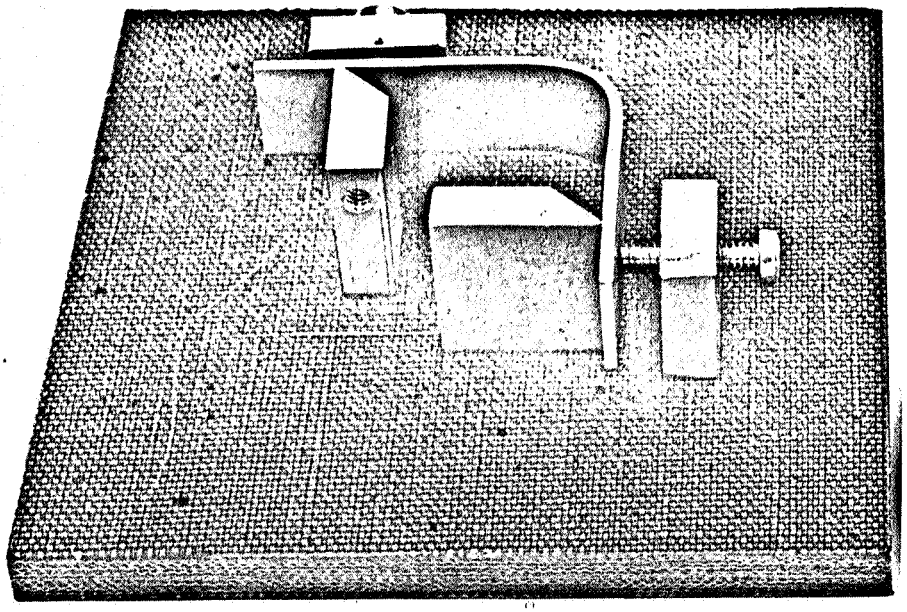


Figure 5. Type B Specimen Mounted in Knife-edge Test Fixture

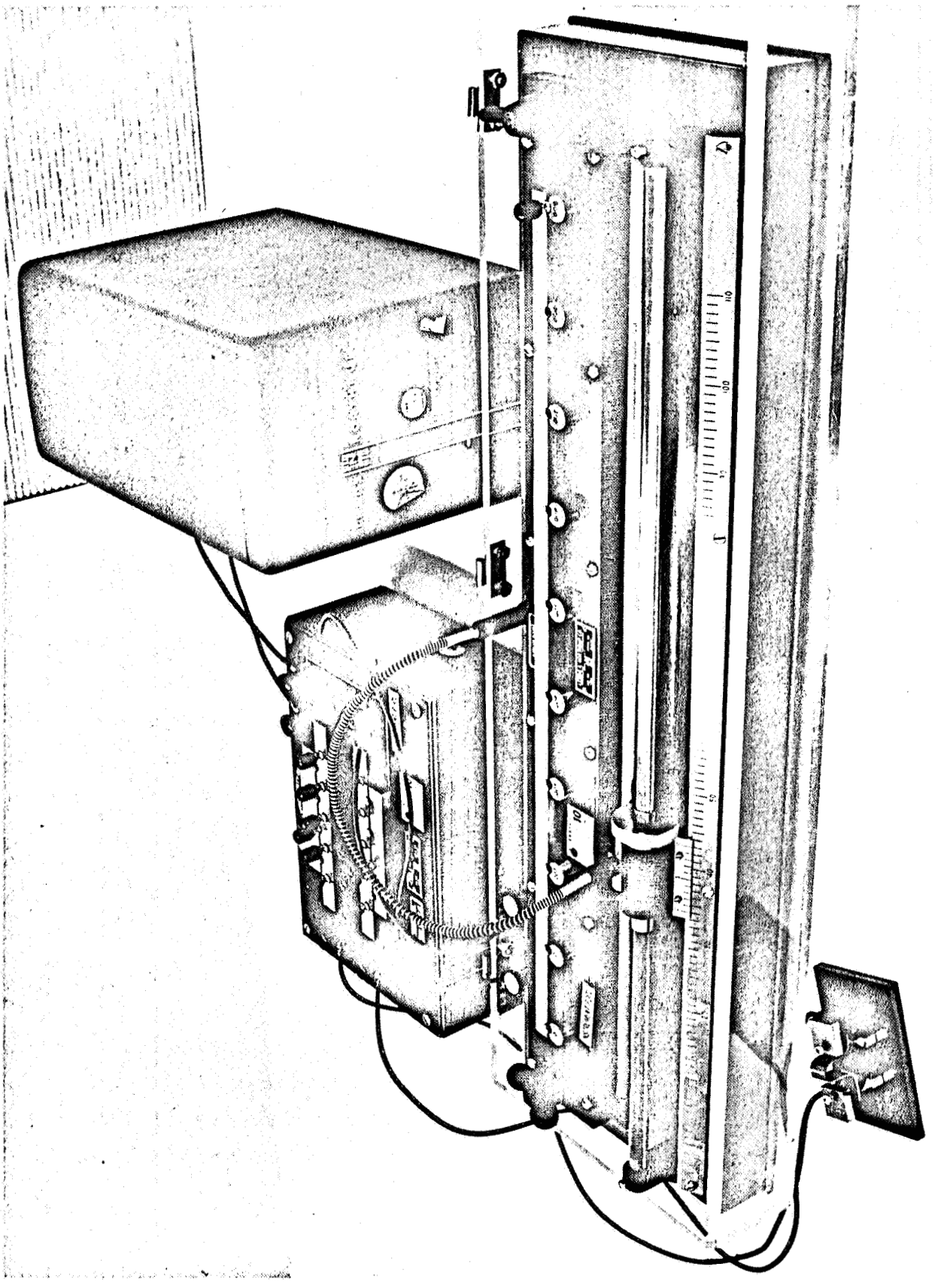


Figure 6. Electrical Resistance Measurement Test Equipment

2.2 EXPERIMENTAL PROCEDURE (Cont.)

The specimens then were stress relieved in laboratory type Marshall or Hoskins tube furnaces. Groups of three each of both the "A" and the "B" types of specimens were stress relieved at 1025°F for periods of 1, 2, 4 and 8 hours, and at 1350°F for periods of 1/2, 1, 2 and 4 hours. Following the re-etching of the electrical contact areas, the electrical resistance of each specimen again was measured and recorded, and the percentage change in resistance was calculated. The data for each group of three specimens then was averaged and plotted as a function of time at the respective temperatures.

During the evaluation of the "A" and "B" specimens, two additional groups of specimens were prepared and tested in order to obtain additional information and data. The first group, consisting of four .50" x 4.00" specimens, was not formed in any manner. The specimens were cut from "as received" material and the electrical resistance of each specimen was measured and recorded. Two of the specimens then were stress relieved at 1025°F, the other two at 1350°F, for cumulative periods of 1/2, 1, 2, 4 and 8 hours, respectively. Following each incremental period, the specimens were etched and the electrical resistance of each specimen was measured (four measurements - each specimen) and recorded. The data for each group of two specimens then was averaged and plotted as a function of time at the respective temperatures.

The second group, consisting of five .50" x 18.00" specimens, was heated at 1350°F for approximately 15 minutes, formed to a 30" radius in a standard LMSC beryllium forming die, and immediately removed from the die and allowed to air cool. The specimens then were stress relieved as follows:

1. No additional heat treatment
2. Stress relieved at 1025°F for 2 hours
3. Stress relieved at 1025°F for 4 hours
4. Stress relieved at 1350°F for 2 hours
5. Stress relieved at 1350°F for 4 hours

2.2 EXPERIMENTAL PROCEDURE (Cont.)

The maximum height of each specimen, above a fixed reference plane, then was determined by means of an optical comparator. This measurement is illustrated in Figure 7. If the specimens contained residual stresses after these treatments, their presence could be determined by etching layers of material from one surface of the specimen and measuring the change, if any, in the curvature. The concave surface of each specimen then was masked and layers of material .001", .002" and .004" thick were cumulatively etched from the convex surface. After each layer was removed, the height of each specimen again was measured and recorded.

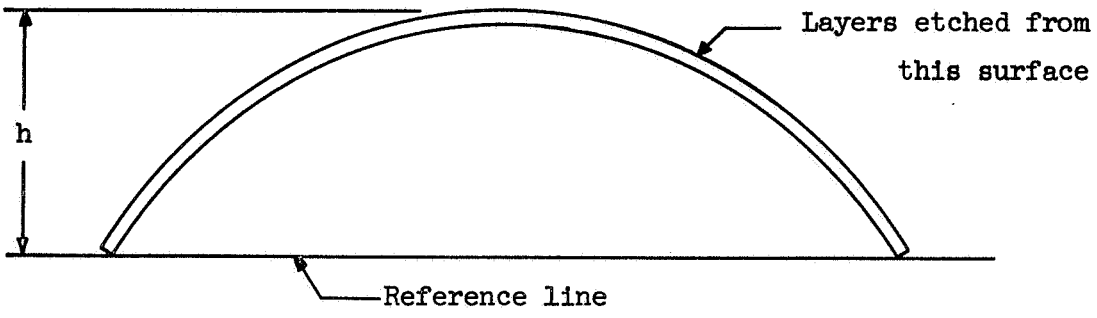


Figure 7. 30" Radius Stress Relief Specimens

All measurements of "h" were corrected for the thickness of material removed during the etching operation, e.g., if .001" was removed and the change in "h" was -.009", the corrected change, -.008", was entered in Table IV.

2.3 RESULTS AND DISCUSSION

Tables II and III present the tabulated electrical resistance data for the Type "A" and "B" specimens, respectively; Figures 8 through 11 present the plotted data. An examination of the tabulated data revealed that the changes in electrical resistance were quite small, ranging from 1×10^{-6} to 68×10^{-6} ohms. The standard deviation, determined by making 10 measurements of a single specimen, was found to be $\pm 16 \times 10^{-6}$ ohms. The application of this standard deviation to each value of percentage change in resistance indicates that each value may vary as much as ± 1.1%. This span is illustrated in the figures.

As clearly shown in Figure 8, the Type "A" specimens exhibited a rapid decrease in electrical resistance, after being stress relieved at 1025°F for one hour, followed by a more gradual decrease thereafter. Even after 8 hours at this temperature, the resistance continued to decrease, possibly indicating the presence of remaining residual stress. Based on the assumption that decreases in resistance could be correlated directly with decreases in residual stress, it was anticipated that stress relieving at 1350°F would result in a more rapid decrease in electrical resistance than occurred during the stress relieving operations at 1025°F. However, as illustrated in Figure 9, a slight increase occurred after 1 to 2 hours, followed by a gradual decrease after 4 hours.

The Type "B" specimens, stress relieved at 1025°F, also exhibited a rapid decrease in electrical resistance after two hours, followed by a more gradual decrease up to 4 hours, and a subsequent "leveling out" thereafter as illustrated in Figure 10. This "leveling out" was believed to be an indication that the residual stresses had been removed after 4 hours at this temperature.

TABLE II

ELECTRICAL RESISTANCE - TYPE "A" SPECIMENS

Specimen Number	Annealing Temperature °F	Annealing Time Hours	Resistance		
			As Formed- ohms($x10^{-4}$)	As Annealed ohms($x10^{-4}$)	Change %
5	1025	1	15.14	14.69	-3.0
6	1025	1	15.02	14.64	-2.5
8	1025	1	14.80	14.45	-2.4
					Avg. -2.6
1	1025	2	15.95	15.71	-1.5
3	1025	2	15.15	14.94	-1.4
4	1025	2	14.97	14.62	-2.3
					Avg. -1.7
13	1025	4	14.68	14.63	-2.2
14	1025	4	14.68	14.28	-2.7
15	1025	4	14.69	14.37	-2.2
					Avg. -2.4
31	1025	8	14.64	13.96	-4.6
32	1025	8	14.51	14.19	-2.2
62	1025	8	15.80	15.10	-4.4
					Avg. -3.7
16	1350	1/2	15.25	15.34	+0.6
17	1350	1/2	15.49	15.50	+0.1
18	1350	1/2	15.30	15.57	+1.8
					Avg. +0.8
24	1350	1	15.08	15.12	+0.3
25	1350	1	15.14	14.90	-1.6
27	1350	1	14.56	14.55	-0.1
					Avg. -0.5
20	1350	2	15.12	15.12	0.0
22	1350	2	14.84	15.02	+1.2
23	1350	2	14.99	15.06	+0.5
					Avg. +0.6
28	1350	4	14.80	14.79	-0.1
29	1350	4	14.41	14.40	+0.1
30	1350	4	14.63	14.42	-1.4
					Avg. -0.5

NOTE: All specimens cut from sheet No. 468A.

TABLE III

ELECTRICAL RESISTANCE - TYPE "B" SPECIMENS

Specimen Number	Annealing Temperature °F	Annealing Time Hours	Resistance		
			As Formed ohms($\times 10^{-4}$)	As Annealed ohms($\times 10^{-4}$)	Change %
36	1025	1	12.24	12.05	-1.6
37	1025	1	11.85	11.93	+0.7
38	1025	1	12.00	11.93	-0.6
					Avg. -0.5
39	1025	2	11.95	11.73	-1.8
40	1025	2	12.08	11.74	-2.8
41	1025	2	12.47	12.17	-2.4
					Avg. -2.3
*42	1025	4	12.55	12.20	-2.8
*43	1025	4	11.80	11.50	-2.5
60	1025	4	11.99	11.60	-3.3
					Avg. -2.9
44	1025	8	11.75	11.43	-2.7
45	1025	8	12.04	11.87	-1.4
46	1025	8	11.96	11.65	-2.6
					Avg. -2.2
47	1350	1/2	12.13	12.26	+1.1
48	1350	1/2	12.05	12.24	+1.6
49	1350	1/2	11.86	11.89	+0.3
					Avg. +1.0
65	1350	1	12.36	12.37	+0.1
50	1350	1	11.96	11.83	-1.1
51	1350	1	11.95	12.03	+0.7
					Avg. -0.1
52	1350	2	12.05	12.12	+0.6
53	1350	2	12.03	12.19	+1.3
54	1350	2	11.96	12.11	+1.3
					Avg. +1.1
55	1350	4	11.91	12.04	+1.1
57	1350	4	11.96	11.99	+0.3
58	1350	4	11.87	11.89	+0.2
					Avg. +0.5

NOTE: *From Sheet No. 447B, all others from Sheet No. 468A.

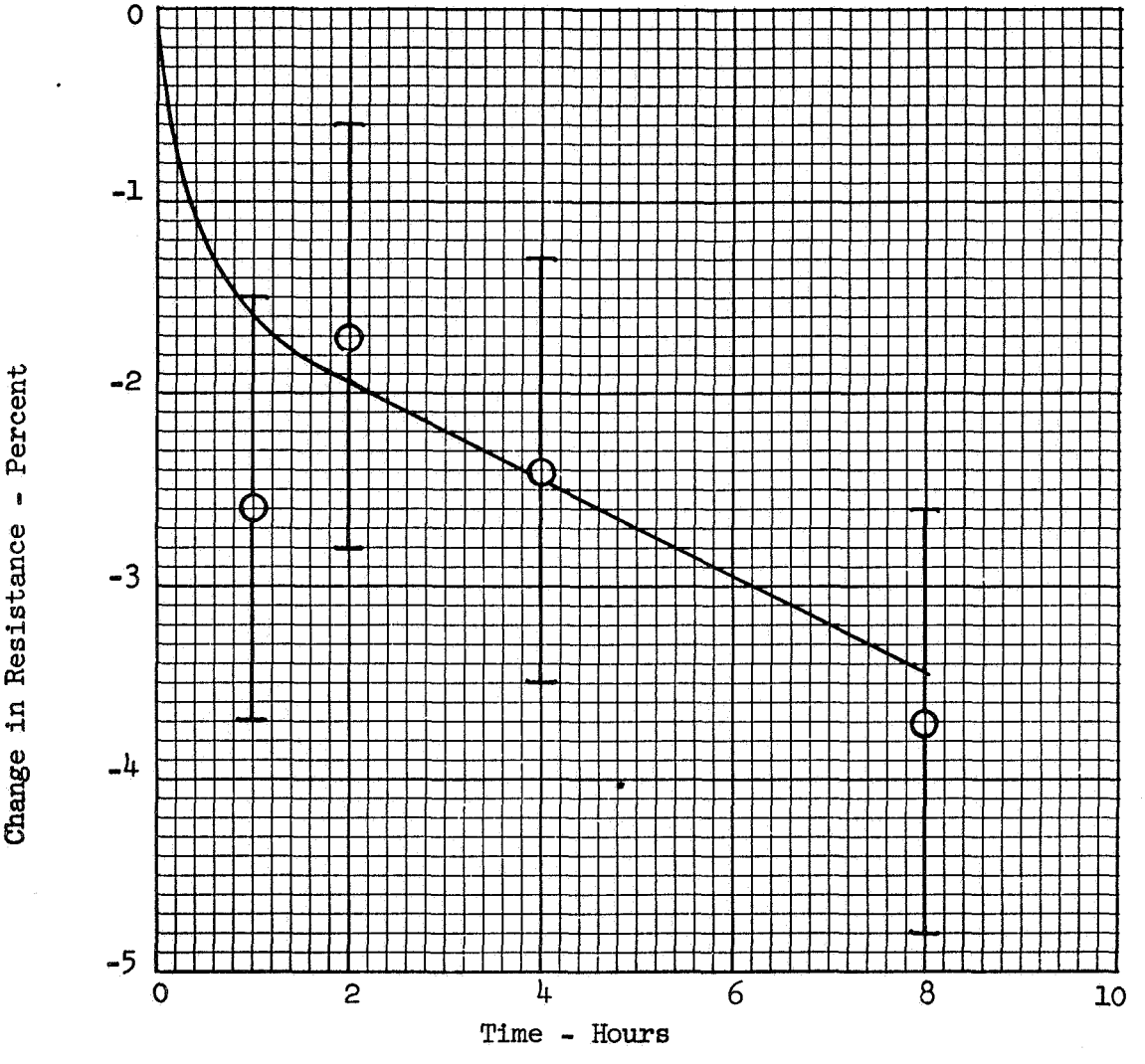


Figure 8. Resistance Change - Type "A" Specimens
Stress Relieved at 1025°F

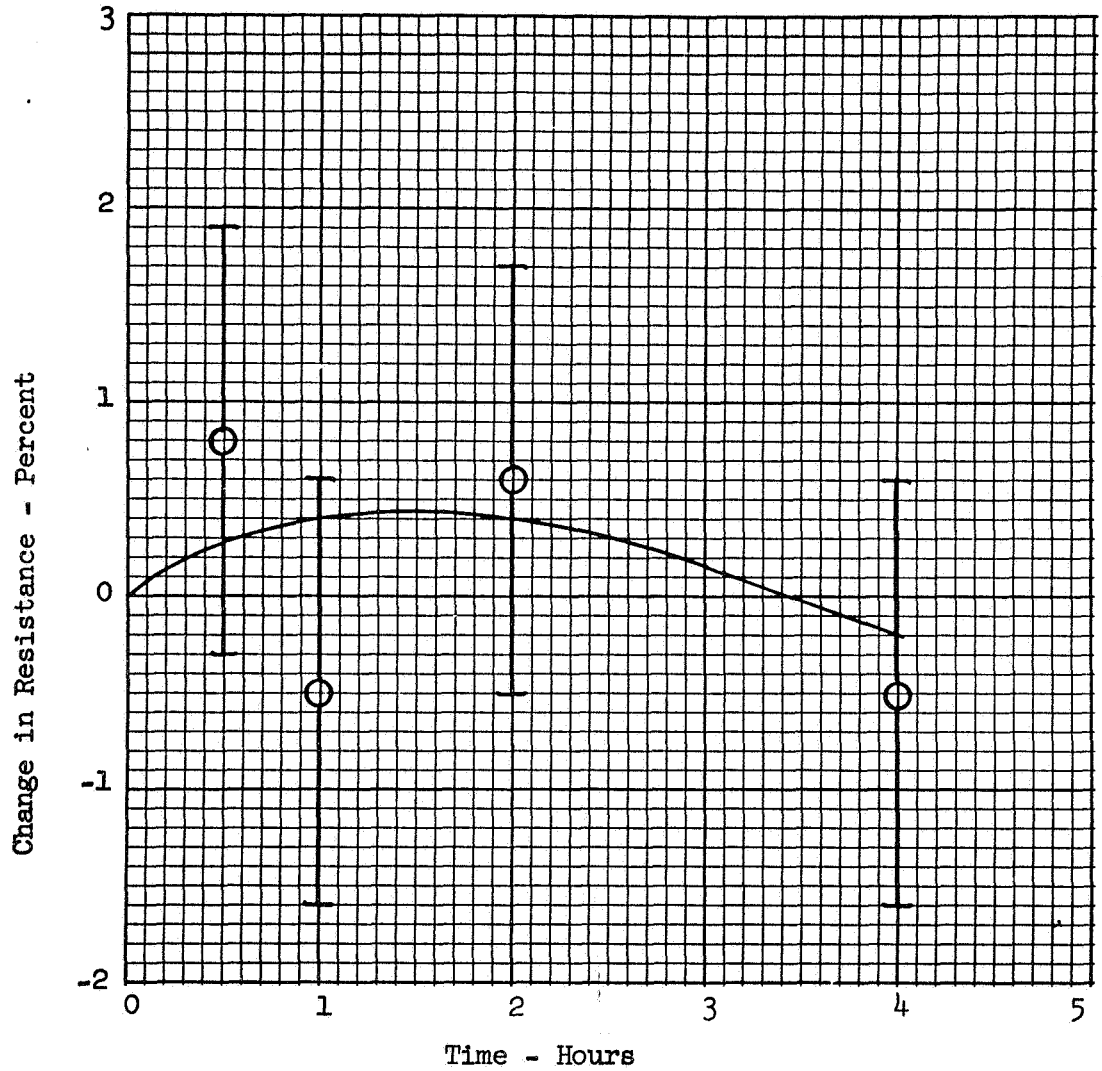


Figure 9. Resistance Change - Type "A" Specimens Stress Relieved at 1350°F

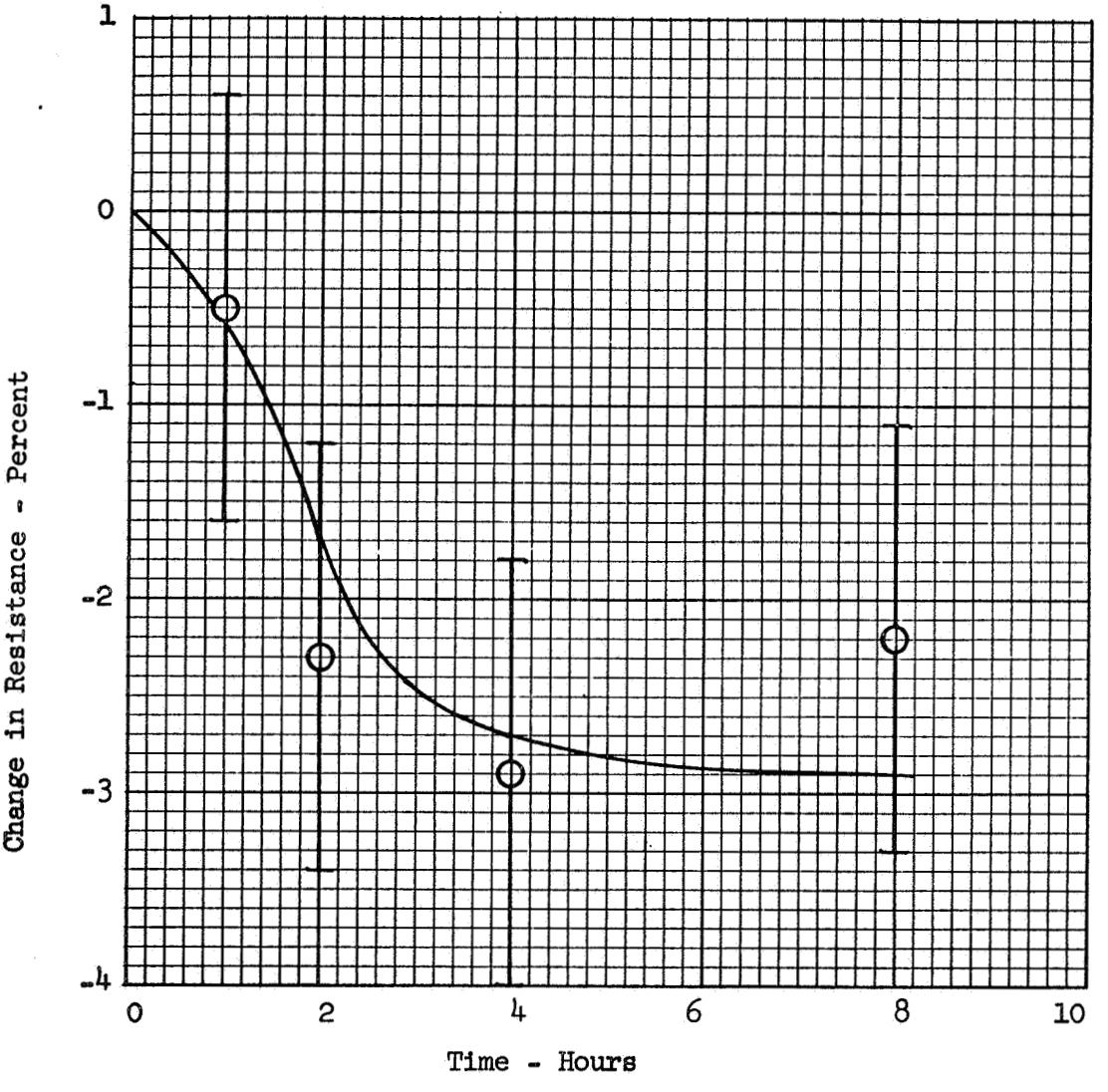


Figure 10. Resistance Change - Type "B" Specimens Stress Relieved at 1025°F

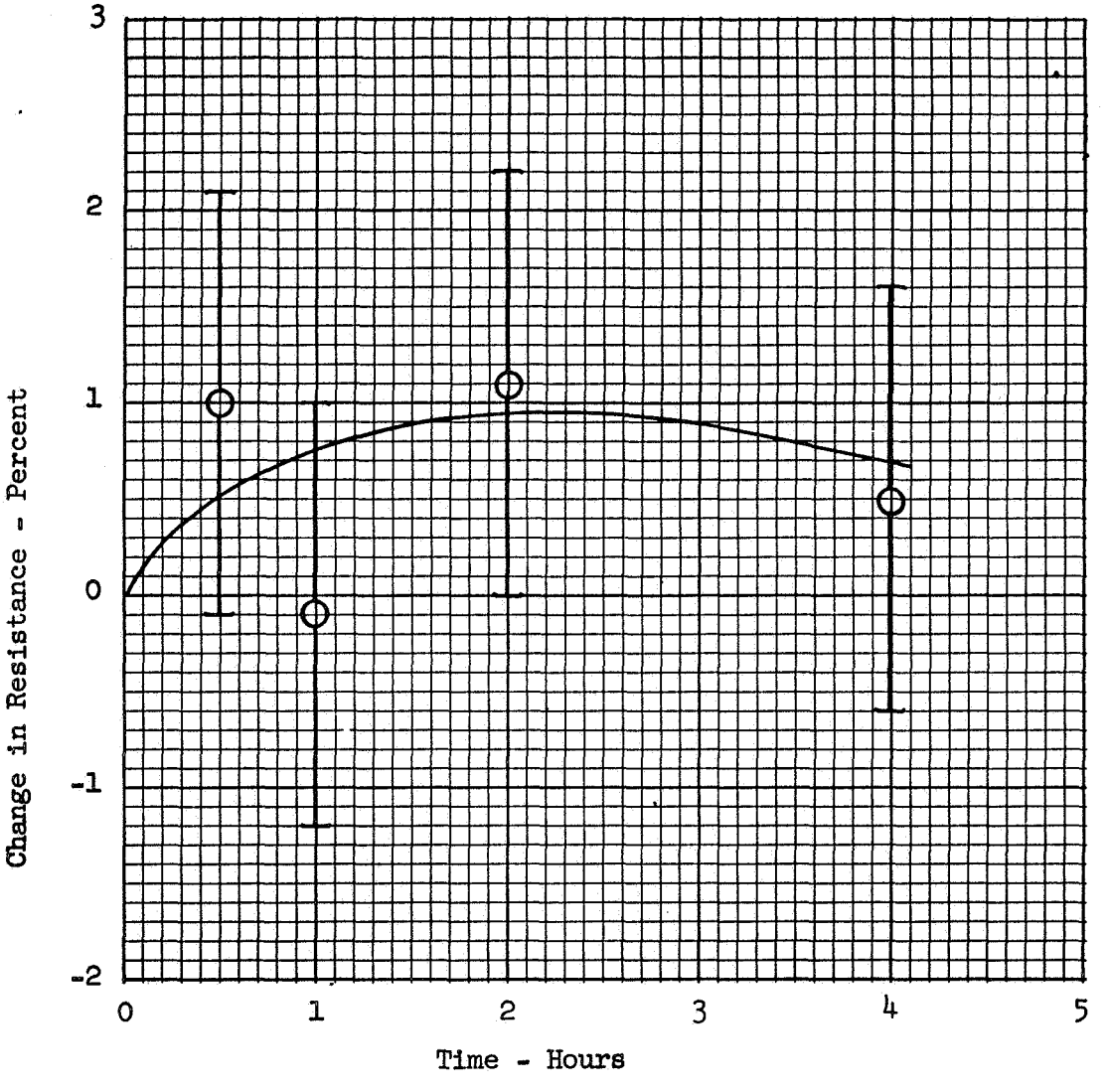


Figure 11. Resistance Change - Type "B" Specimens Stress Relieved at 1350°F

2.3 RESULTS AND DISCUSSION (Cont.)

As illustrated in Figure 11, the Type "B" specimens stress relieved at 1350°F also exhibited an initial increase in resistance, followed by a gradual decrease, very similar to that exhibited by the Type "A" specimens.

The interpretation of the relative behavior of the specimens stress relieved at 1025°F and 1350°F is difficult. If the changes in electrical resistance can be attributed solely to the relief of residual stresses, then the specimens stress relieved at 1350°F should have exhibited greater decreases in resistance than those stress relieved at 1025°F. For a given period of time, treatment at a higher temperature normally will result in a correspondingly greater decrease in residual stress. The fact that this relationship was not observed indicated the occurrence of some other phenomenon. The exact nature of this phenomenon is unknown, although it is believed to be related to the impurities present in the beryllium, primarily iron, that either are being taken into solution or are being precipitated as compounds.

The investigation of the effects of stress relieving the .50" x 4.00" unformed specimens then was initiated in order to obtain additional data. Table IV presents the tabulated electrical resistivity data; Figure 12 presents the plotted data. This data is presented in terms of change in resistivity, rather than change in resistance, as the same specimens were used throughout the investigation. The etching of the specimens, subsequent to each stress relieving period, resulted in dimensional changes of such magnitude that the calculation of the actual resistivity was required in each case. The resistivity was calculated as follows:

2.3 RESULTS AND DISCUSSION (Cont.)

$$\rho = \frac{RA}{L}$$ where:

ρ = Resistivity; microhm-inches
R = Resistance; microhms
A = Cross-section area; square inches
L = Length between contact points; inches

An examination of the plotted data revealed that the specimens stress relieved at 1025°F exhibited a rapid decrease in electrical resistivity after 1 hour, followed by a more gradual decrease thereafter. The specimens stress relieved at 1350°F exhibited a rapid initial decrease in electrical resistivity after 1/2 hour, followed by an increase after 1 hour, and a gradual decrease thereafter. If the first 1/2 hour is omitted, the trends in the changes in resistance were very similar to those exhibited by the Type "A" and "B" specimens stress relieved in a similar manner. It may be assumed that the first 1/2 hour corresponds to the time required for the heating and forming of the Type "A" and "B" specimens, and that similar changes occurred in these specimens during the heating and forming operations.

The results obtained from the tests of these unformed specimens are similar to those obtained by Wolff, Gelles, and Aronin* during tests of extruded commercially pure beryllium; they observed decreases in electrical resistance in specimens annealed at 1110°F and 1470°F for periods of time up to 800 hours. These continued decreases were attributed to the presence of iron in solid solution precipitating as the compound $FeBe_5$.

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*A. K. Wolff, S. H. Gelles and C. R. Aronin, "Impurity Effects in Commercially Pure Beryllium," The Physical Metallurgy of Beryllium, Institute of Metals, London, 1963.

TABLE IV

ELECTRICAL RESISTIVITY - UNFORMED SPECIMENS

Specimen Number	Annealing Temperature °F	Annealing Time Hours	Resistance ohms(x10^{-5})	Resistivity microhm-in (x10^{-7})	Resistivity Change %
AR-1	As Rec'd	0	10.3	6.40	0
AR-2	As Rec'd	0	10.3	6.39	0
					Avg. 0
AR-1	1025	1/2	10.2	6.33	-1.1
AR-2	1025	1/2	10.2	6.32	-1.1
					Avg. -1.1
AR-1	1025	1	9.9	6.15	-3.9
AR-2	1025	1	9.9	6.13	-4.1
					Avg. -4.0
AR-1	1025	2	9.6	5.96	-6.9
AR-2	1025	2	9.6	5.95	-6.9
					Avg. -6.9
AR-1	1025	4	9.5	5.90	-7.8
AR-2	1025	4	9.5	5.88	-8.0
					Avg. -7.9
AR-1	1025	8	9.3	5.72	-10.6
AR-2	1025	8	9.0	5.56	-13.0
					Avg. -11.8
AR-3	As Rec'd	0	9.9	6.22	0
AR-4	As Rec'd	0	9.9	6.39	0
					Avg. 0
AR-3	1350	1/2	9.4	5.90	-5.2
AR-4	1350	1/2	9.6	5.90	-7.7
					Avg. -6.5
AR-3	1350	1	10.5	6.02	-3.2
AR-4	1350	1	9.9	6.08	-4.9
					Avg. -4.1
AR-3	1350	2	10.5	6.02	-3.2
AR-4	1350	2	9.8	6.02	-5.8
					Avg. -4.5
AR-3	1350	4	10.3	5.91	-5.0
AR-4	1350	4	9.8	6.02	-5.8
					Avg. -5.4
AR-3	1350	8	8.7	4.98	-20.0
AR-4	1350	8	8.9	5.48	-14.2
					Avg. -17.1

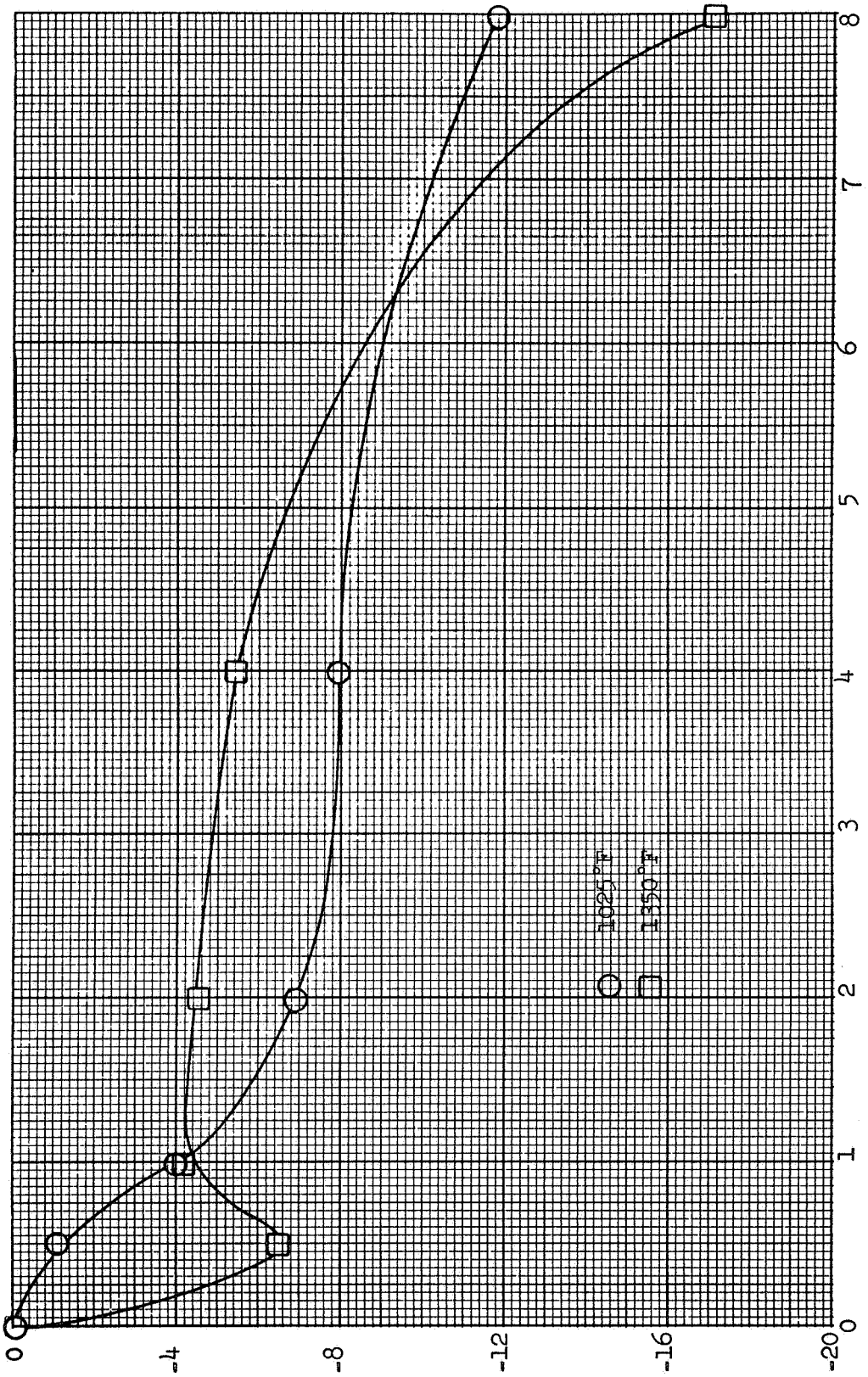


Figure 12. Resistivity Change - Unformed Specimens Stress Relieved at 1025°F and 1350°F

2.3 RESULTS AND DISCUSSION (Cont.)

On the basis of these results, it was believed that, although some stress relief undoubtedly did occur during the treatment of the Type "A" and "B" specimens, the aging effects that occurred simultaneously tended to overshadow the stress relief effects.

The testing of the fourth group of specimens then was initiated in an effort to separate the stress relieving effects from the aging effects. Table V presents the tabulated change in height data for the 30" radius curved specimens.

TABLE V

CURVATURE CHANGES - 30" RADIUS SPECIMENS

Annealing Temperature °F	Annealing Time Hours	Change in Height "h" after etching Total Thickness of Material Removed - inch		
		.001	.002	.004
As received		-.008	-.006	-.002
1025	2	+.001	-.008	-.001
1025	4	-.003	+.003	-.001
1350	2	-.006	+.027	-.035
1350	4	-.013	-.001	-.002

The results of these stress relieving tests indicate that the treatments used did not result in the complete removal of the stresses. In all cases, the changes in the height "h", observed after each etching operation, indicate the presence of some remaining residual stress in all of the specimens. If no changes had occurred, the successful removal of all residual stresses could have been concluded.

The specimen stress relieved at 1025°F for 4 hours exhibited the least change in curvature. Therefore, for this specific forming operation, stress relief at 1025°F for 4 hours appears to be the better treatment. This result, however, should not be generalized as each type of

2.3 RESULTS AND DISCUSSION (Cont.)

forming operation, depending upon its severity, may require a specific time and temperature cycle for the relief of all residual stresses.

In conclusion, the reduction of residual stresses noted during this brief investigation, measured by means of the changes in electrical resistivity and the changes in curvature, indicates that the nominal thermal treatment conditions for the relief of residual stresses appear to be 1025°F for approximately 4 hours. However, the optimization of the stress relief procedure for general application would require extensive basic metallurgical research and analysis which is not believed to be within the scope of this study.

SECTION 3.0
COOLING RATE

Current production forming procedures require uniform cooling of the entire part subsequent to the forming operation in order to prevent the formation of undesirable stress patterns and subsequent warpage. The effects of rapid quenching subsequent to hot forming at 1350°F, therefore, have received little attention. A limited exploratory investigation of these effects, and an evaluation of the possible applicability of rapid cooling to production operations, therefore, was included in this program.

3.1 INTRODUCTION

The object of this phase of the program was the determination of the effects of various quenching rates on the linear dimensions, material contour (warpage), and mechanical properties; and the detection of any significant trends.

Due to the preliminary nature of this study, only a limited investigation was contemplated. Three quenching mediums were employed:

a. Ambient air at 75°F
b. Oil at 88°F
c. Deep freeze at -85°F

Table VI presents the certified chemical analyses and mechanical properties of the two lots of material procured for this investigation from the Brush Beryllium Company and The Beryllium Corporation in accordance with the requirements of Lockheed Specification, LAC 07-4008.

TABLE VI

CHEMICAL ANALYSES AND MECHANICAL PROPERTIES - COOLING RATE SPECIMENS

VENDOR DATA

Material Identification

Vendor	Brush Beryllium Co.	The Beryllium Corp.
Lot. No.	9967	397D
Sheet No.	447B	HR 33-1
Gage	.060	.060

Chemical Analyses - %

Be Assy	98.29	98.22
Be O	1.66	1.91
Fe	.138	.134
Si	.050	.050
Al	.095	.083
Mg	.011	.027
Mn	.010	(1)
Ni	.021	(1)
Cr	.012	(1)
C	.120	.102

Mechanical Properties

Sheet No.	Test Direction	Ftu Psi	Fty Psi	Elong. % in 1"
447B	L	78,300	57,400	11.5
447B	T	86,200	58,100	14.0
HR 33-1	L	70,600	51,200	8.0
HR 33-1	T	78,400	53,000	21.0

(1) .04 maximum

3.2 EXPERIMENTAL PROCEDURE

Three 5.00" x 10.00" specimens were cut, using the abrasive wheel technique, from the two lots of material. Two specimens were cut from sheet number HR 33-1; the third from sheet number 447B. Each specimen then was permanently identified, a 1.00 inch grid pattern was lightly scribed on one side, and a .75 inch wide control tensile specimen was cut (abrasive wheel technique) from one end. Figure 13 illustrates the three specimens prior to thermal treatment.

Prior to the initiation of the testing operations, basic dimensions were carefully determined and recorded. Linear measurements, to be used in the determination of dimensional change, were made along the grid lines; a 12-inch Brown and Sharpe vernier caliper was used to insure the accuracy of the measurements. The specimens then were placed on a surface plate and the deviations from "flat" were measured at the grid line intersections by means of a Starrett Dial Indicator.

The three specimens then were heated in an electric furnace at 1350°F for 30 minutes prior to being cooled at the three different rates. The temperature was monitored and recorded with a Brown Strip Recorder. The first specimen, number 6-2-13-01, was allowed to cool in ambient air; the second, number 6-2-13-02, was quenched in oil at 88°F; and the third, number 6-2-13-03, was placed in a Deepfreeze cabinet at -85°F.

The basic dimensional and "flatness" measurements then were repeated, utilizing the same procedures and equipment, to provide comparative data for analysis and evaluation. Following the completion of the second series of measurements, three tensile coupons were cut from each of the three test specimens. The drill jig, illustrated in Figure 21, and the standard router fixture, illustrated in Figure 14, were used in drilling the loading pin holes and cutting the specimens to tensile coupon configuration. The versatility of this LMSC-owned

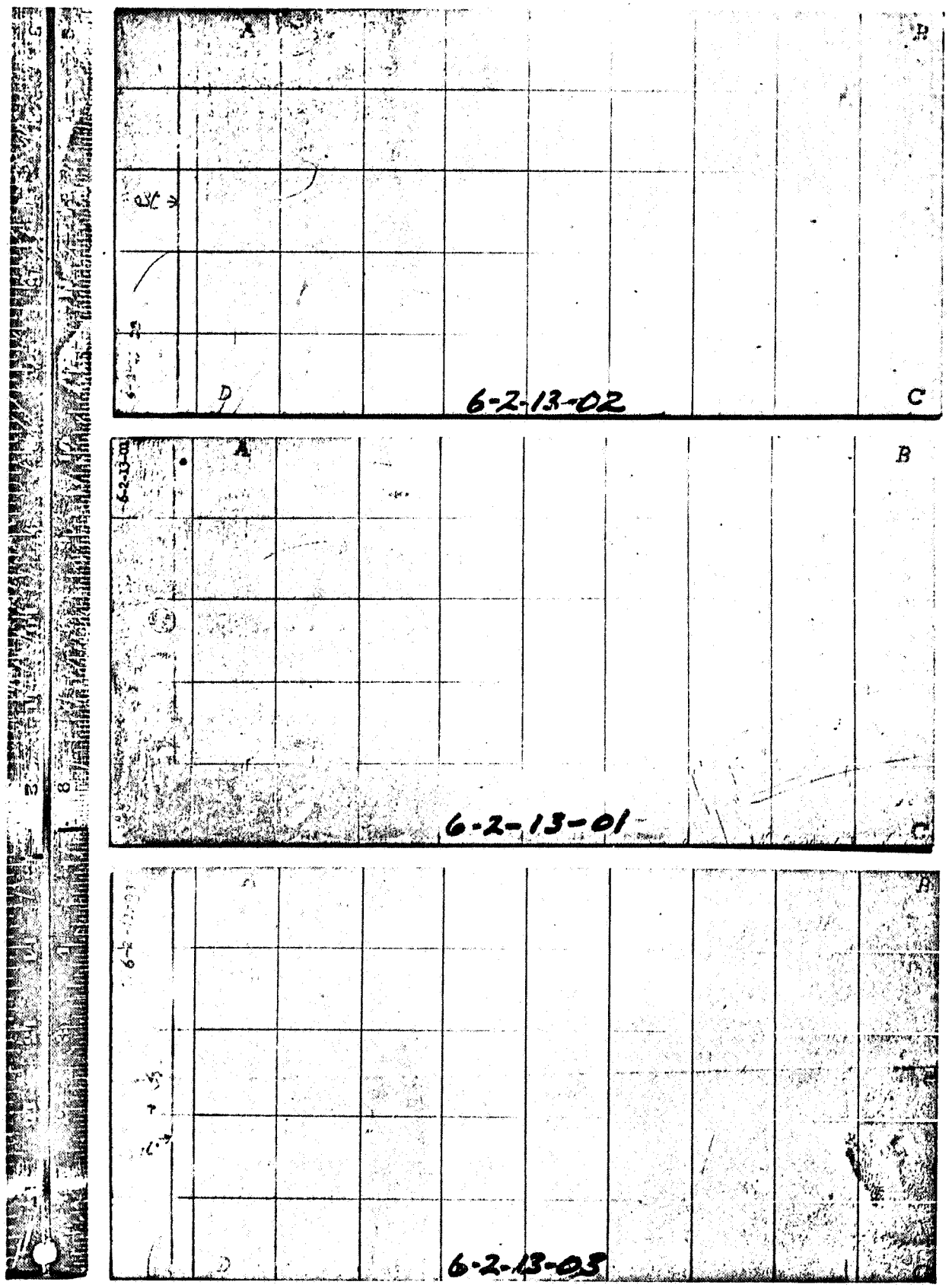


Figure 13. Cooling Rate Specimens - Prior to Thermal Treatment

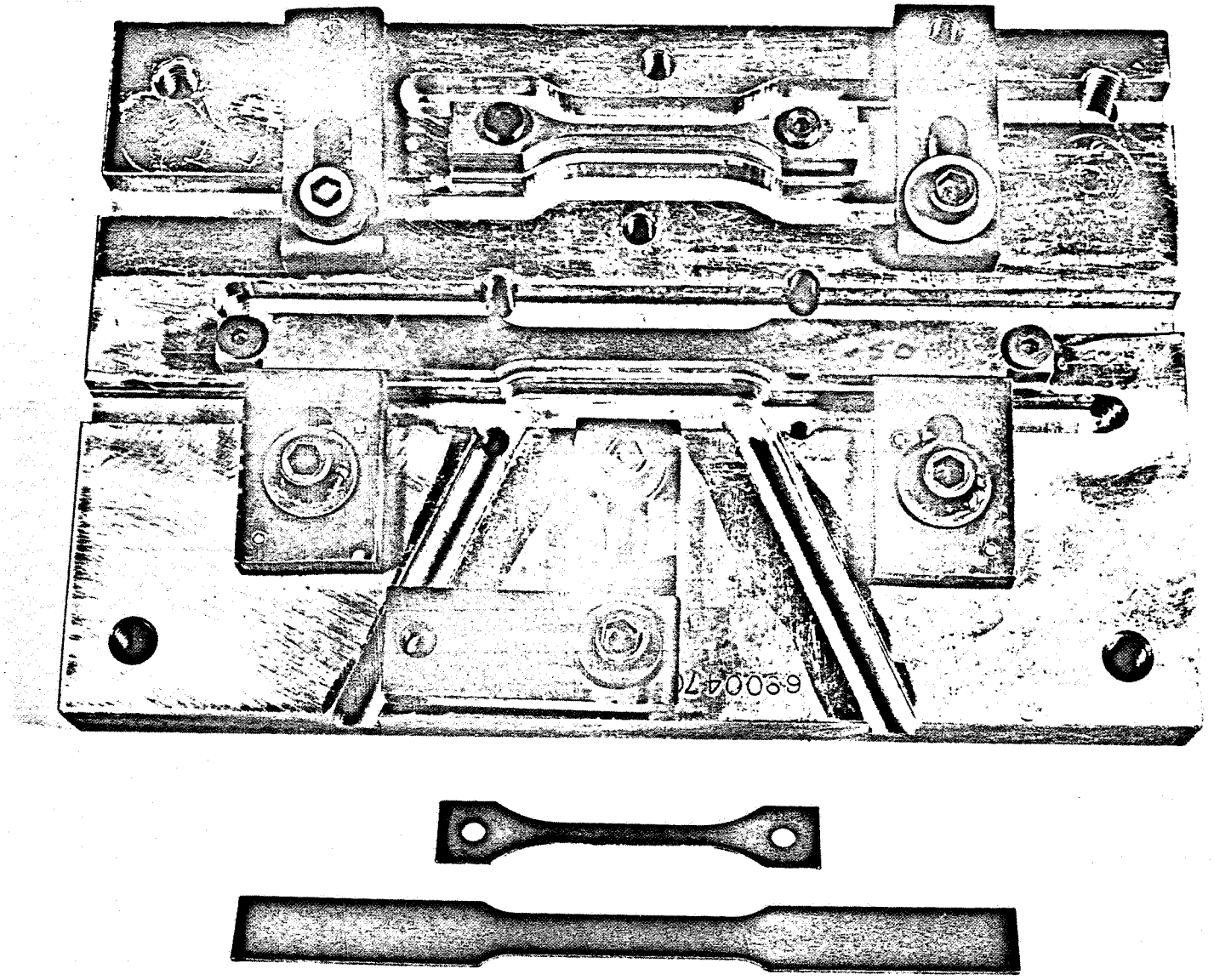


Figure 14. Standard Router Fixture for Preparing Tensile Specimens

3.2 EXPERIMENTAL PROCEDURE (Cont.)

router fixture is clearly illustrated in Figure 14; it may be used to prepare either 1/4" x 1" or 1/2" x 2" gage section tencile specimens. The final preparation, prior to testing, consisted of light abrasive polishing followed by chemical etching.

The tensile tests were performed on a 20,000-pound capacity Riehle Model FS-20 universal testing machine. A Riehle Model DN-10 extensometer was used to measure the strain in the specimens. The specimens were loaded to failure at a strain rate of .005/inch/inch/minute through yield. The charts of the autographically recorded loads were used to determine the load at 0.2% offset and at failure. The total elongation was determined by fitting the fractured specimens together and measuring the distance between the gage marks.

Due to the unexpectedly low mechanical properties exhibited by the "mechanically routed" tensile specimens, including the "as received condition" control specimens, three additional tensile specimens were cut from each of the three "cooling rate" test plates, using the abrasive wheel technique, and were finish machined using the EDM (Electrical Discharge Machining) process. This procedure is described in Section 4.2, and the equipment and typical specimens are illustrated in Figure 21. This second set of specimens was tested in the same manner and with the same equipment as the first set.

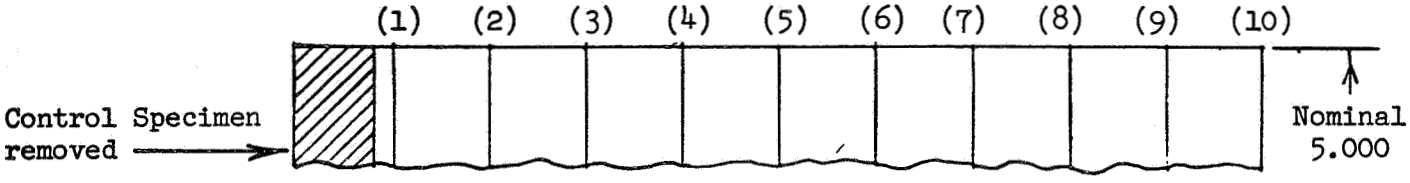
3.3 RESULTS AND DISCUSSION

3.3.1 Dimensional Changes

Table VII presents the tabulated dimensional change data for the ambient air-cooled, the oil-quenched, and the deepfreeze-chilled "cooling rate" specimens, respectively. The original dimensions, the post thermal treatment dimensions, and the net changes are included. Due to the removal of the tensile control specimens from the end of each of the cooling rate specimens, only the width dimensions could be compared.

TABLE VII

DIMENSIONAL CHANGES - COOLING RATE SPECIMENS



Type Cooling	Ambient Air @ 75°F			Oil Quench @ 88°F			Deepfreeze @ -85°F		
Specimen No.	6-2-13-1			6-2-13-2			6-2-13-3		
Position	Original	Post Th.Tr.	Net Change	Original	Post Th.Tr.	Net Change	Original	Post Th.Tr.	Net Change
1	5.0055	5.0060	+.0005	5.0000	5.0005	+.0005	5.0250	5.0265	+.0015
2	5.0060	5.0065	+.0005	5.0000	5.0005	+.0005	5.0250	5.0275	+.0025
3	5.0072	5.0080	+.0008	5.0000	5.0005	+.0005	5.0270	5.0280	+.0010
4	5.0083	5.0090	+.0007	5.0000	4.9995	-.0005	5.0270	5.0285	+.0015
5	5.0100	5.0105	+.0005	5.0000	4.9995	-.0005	5.0290	5.0305	+.0015
6	5.0120	5.0130	+.0010	5.0000	5.0005	+.0005	5.0300	5.0315	+.0015
7	5.0150	5.0150	.0000	5.0000	4.9975	-.0025	5.0340	5.0345	+.0005
8	5.0180	5.0180	.0000	5.0000	4.9975	-.0025	5.0360	5.0365	+.0005
9	5.0200	5.0205	+.0005	5.0000	4.9960	-.0040	5.0370	5.0380	+.0010
10	5.0220	5.0235	+.0015	5.0000	4.9960	-.0040	5.0390	5.0380	-.0010
Average Change			+.0006			-.0012			+.0011

3.3.1 Dimensional Changes (Cont.)

The evaluation of the tabulated data indicated that, although slight dimensional changes did occur, the average changes of approximately .01-.02% were too small to be considered significant.

In conclusion, even though the measurements are subject to the normal interpretive error (visual acuity of the personnel making the measurements), the dimensional changes due to rapid cooling are considered to be negligible for all except very large or extremely precise parts and, therefore, normally may be ignored.

3.3.2 Contour Changes (Warpage)

In order to permit ready comparison of the data and to simplify the evaluation of the effects of rapid cooling on the "flatness" of the specimens, each set of data was plotted on an individual "contour map" to pictorially present the "as received" and the "quenched" conditions of the specimens. Figures 15 through 20 present these graphic illustrations.

Visual examination and comparison of the two illustrations of each specimen very clearly show the effect of the various quenching mediums on the original contour of the sheet material. The least waviness or warpage occurred in the air-cooled specimen; in fact, the "flatness" actually was improved; the most severe warpage occurred in the oil-quenched specimen., Figures 15 and 16 clearly show the improvement in the contour of the air-cooled sheet. The deleterious effect of the rapid oil-quench is illustrated in Figures 17 and 18, and the more moderate effect of the deepfreeze-chilling is shown in Figures 19 and 20.

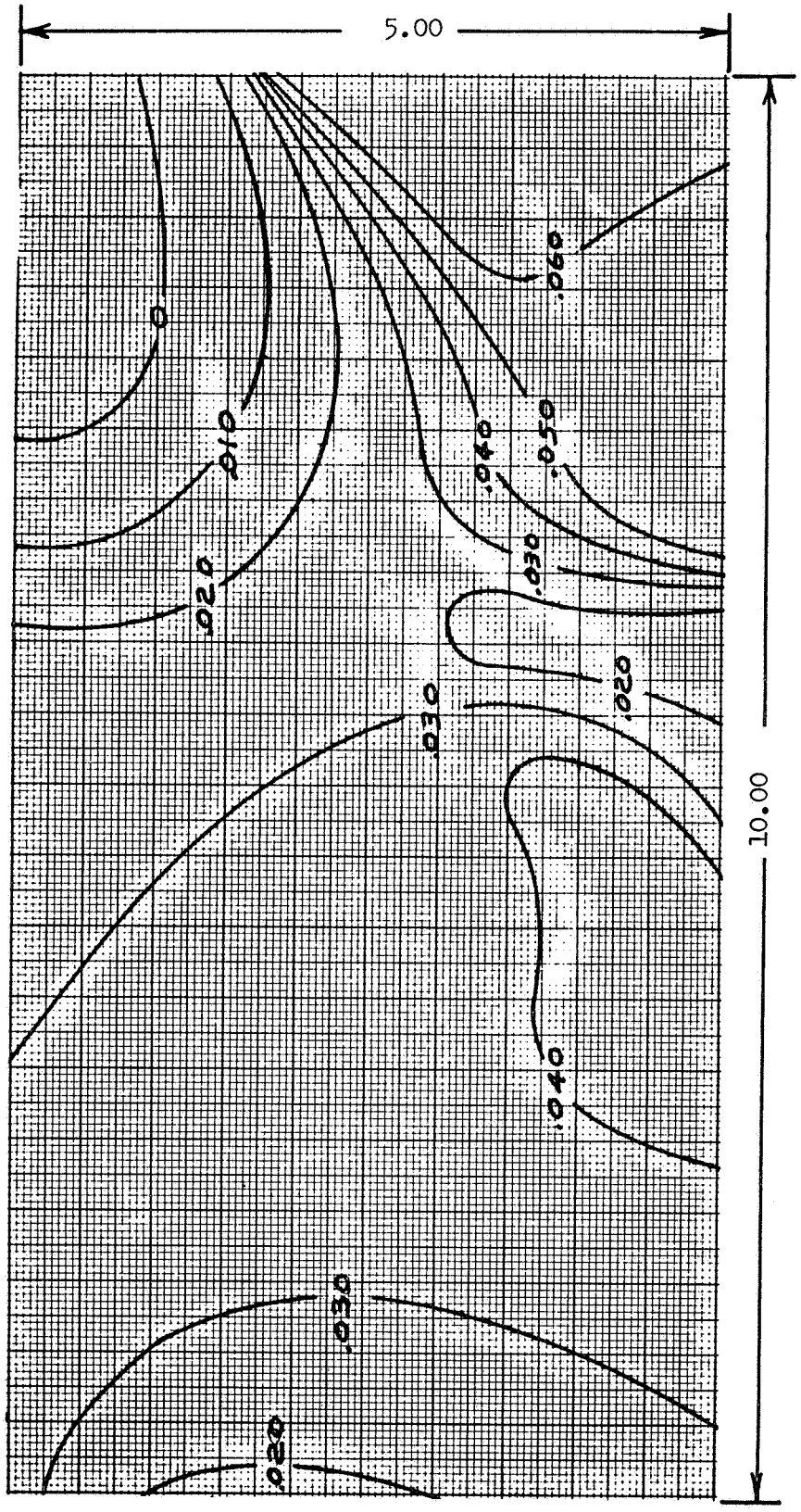


Figure 15. Contour Lines of Specimen No. 6-2-13-01 Prior to Thermal Treatment

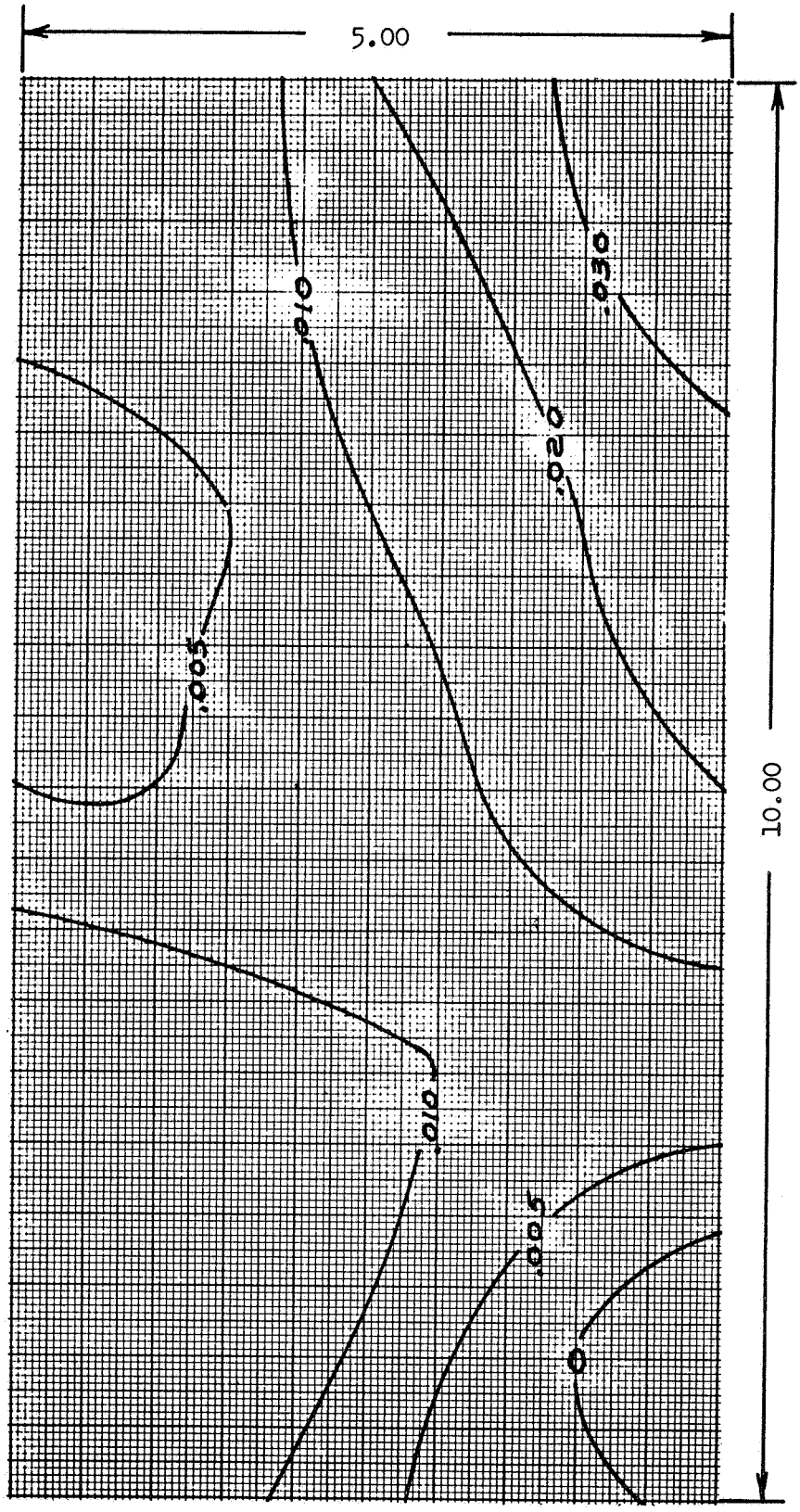


Figure 16. Contour Lines of Specimen No. 6-2-13-01 after Thermal Treatment and Air-Cooling

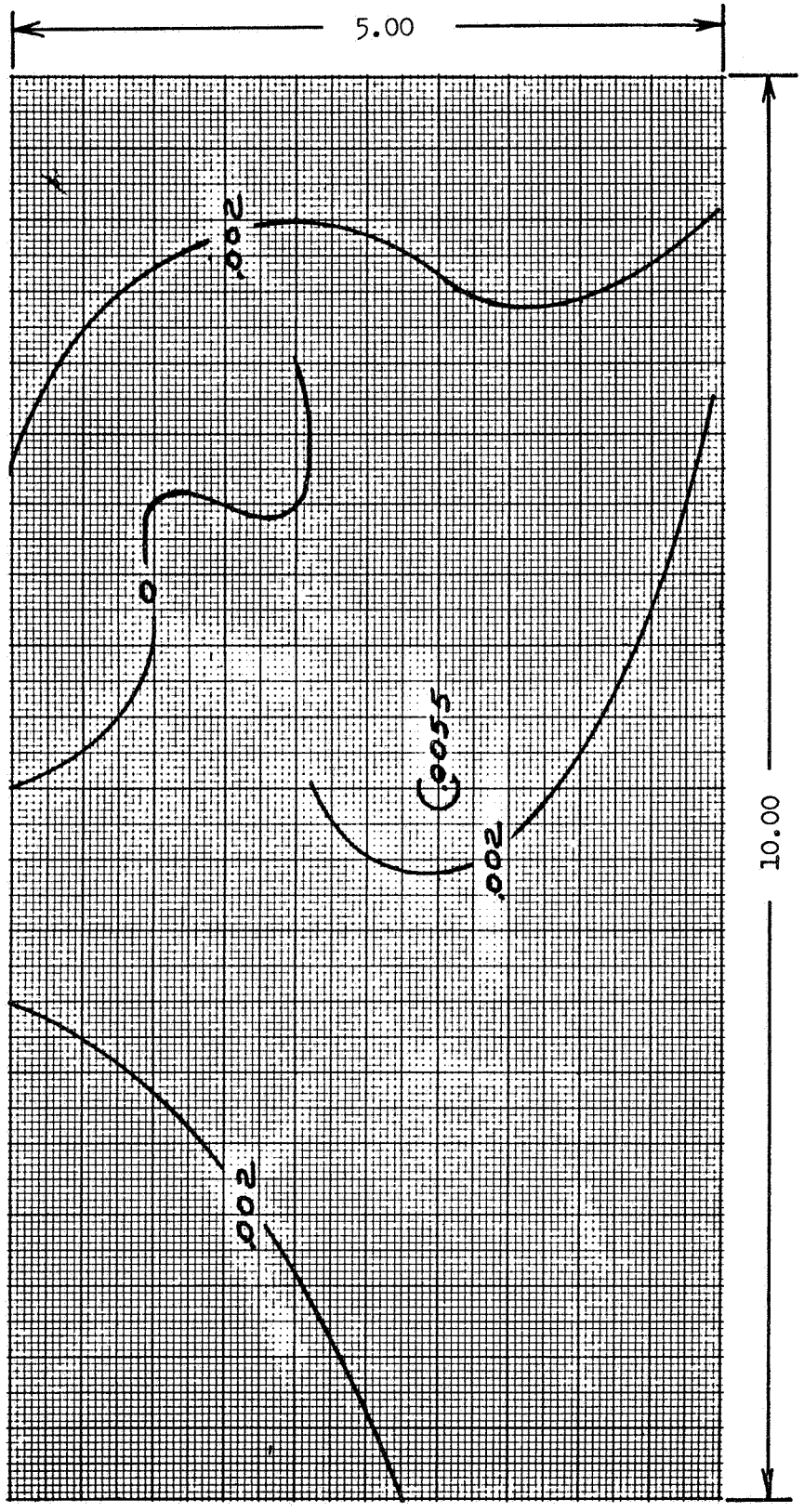


Figure 17. Contour Lines of Specimen No. 6-2-13-02 Prior to Thermal Treatment

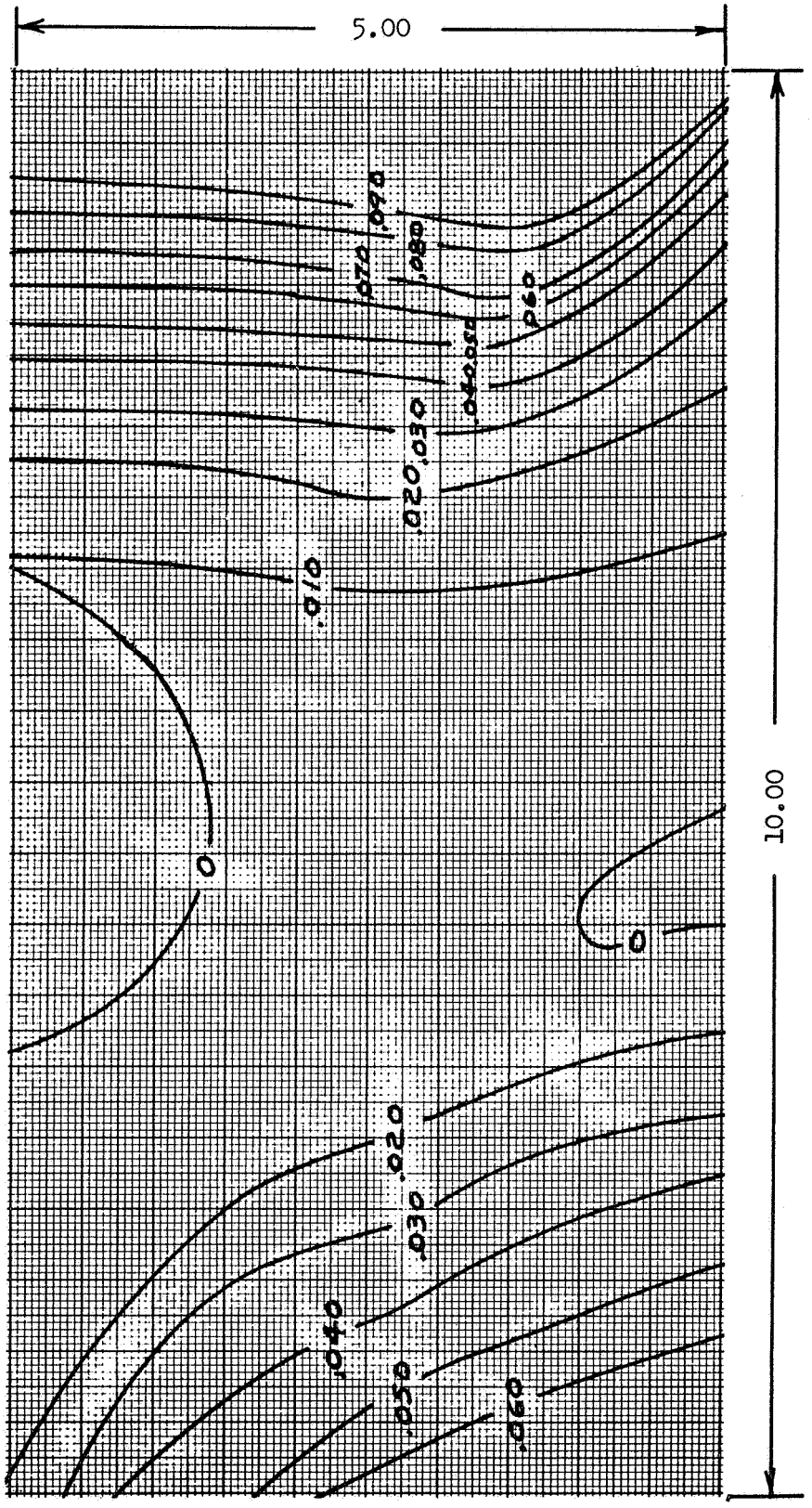


Figure 18. Contour Lines of Specimen No. 6-2-13-02 after Thermal Treatment and Oil Quench

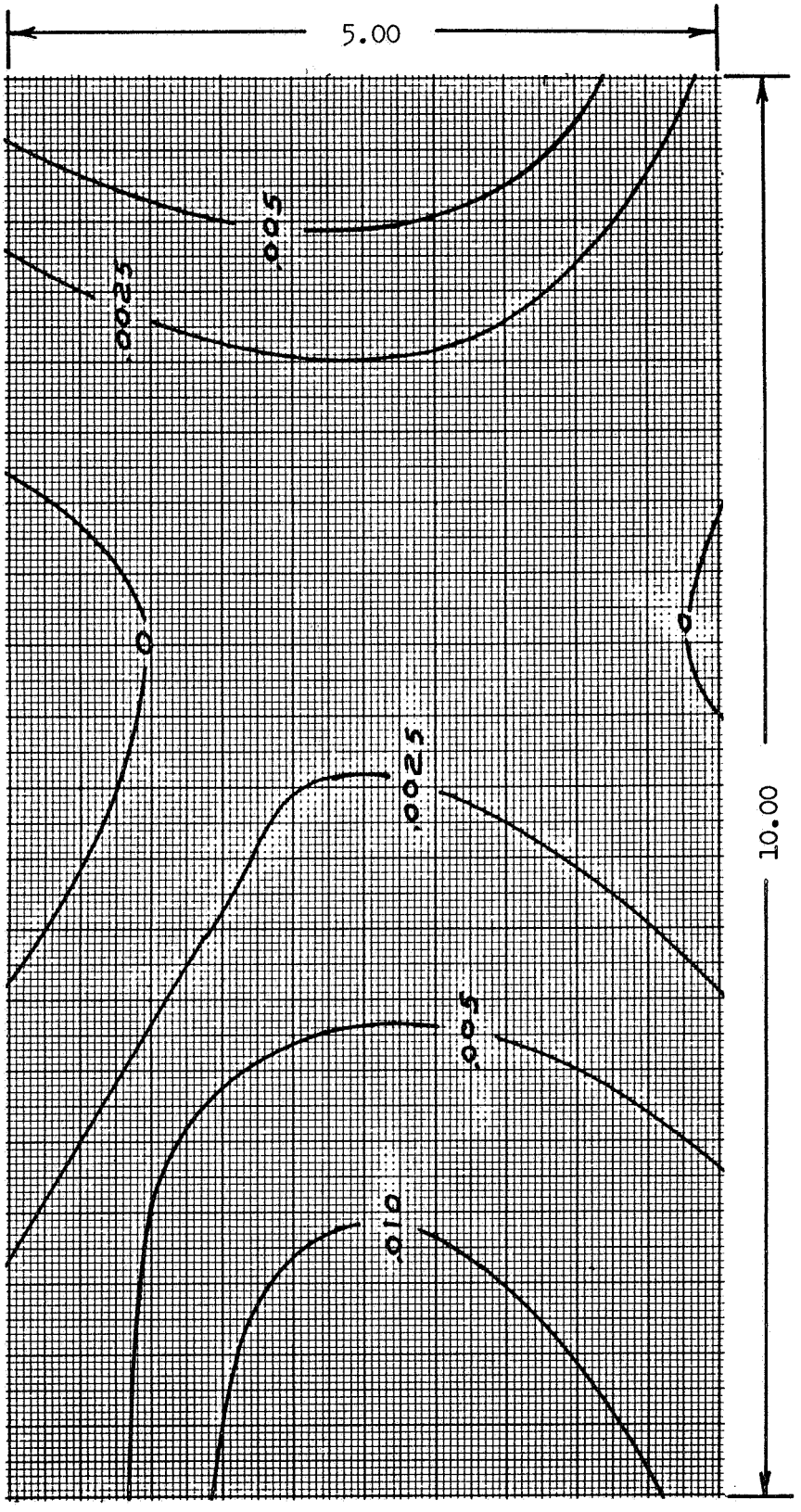


Figure 19. Contour Lines of Specimen No. 6-2-13-03 Prior to Thermal Treatment

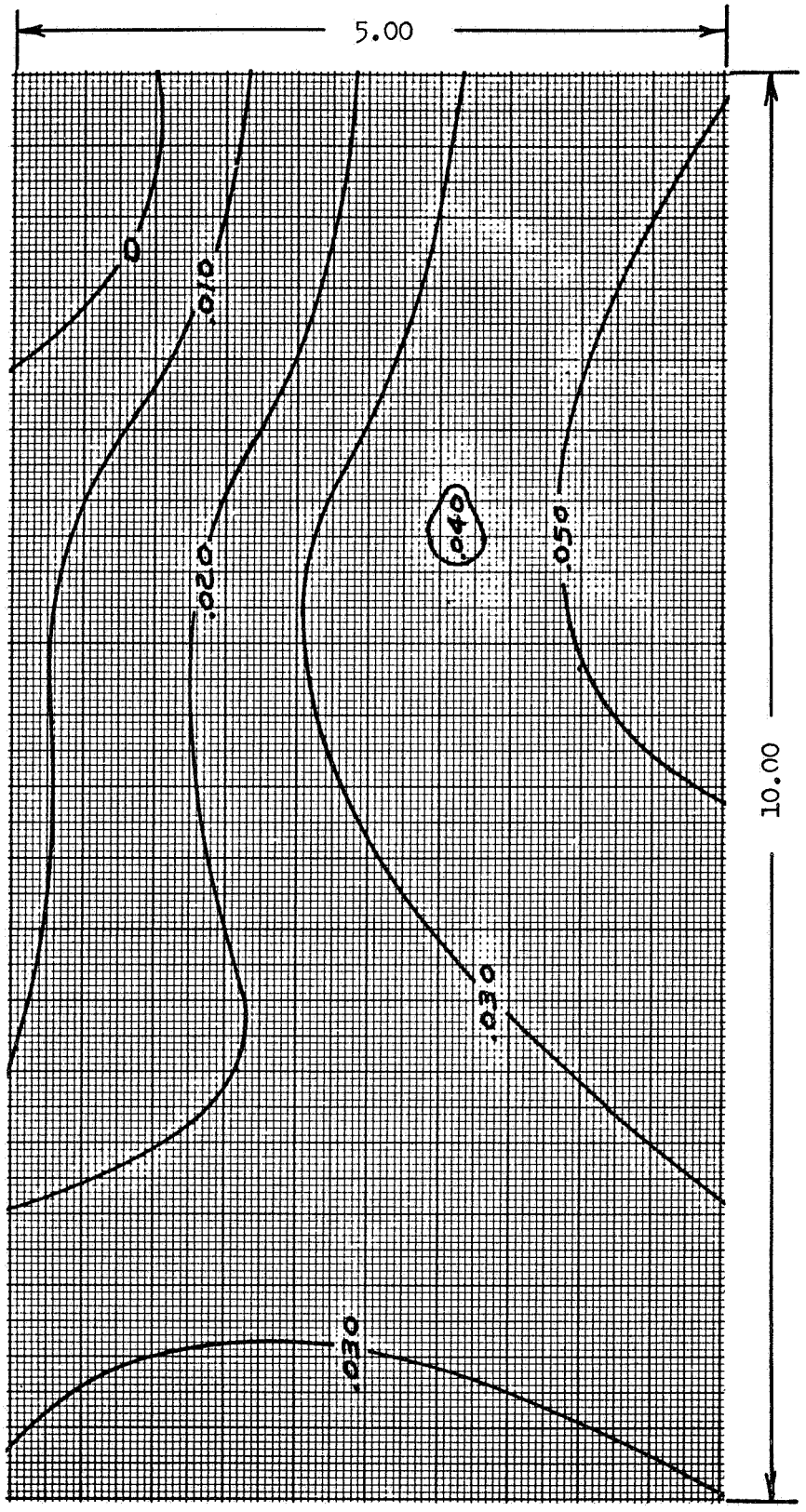


Figure 20. Contour Lines of Specimen No. 6-2-13-03 after Thermal Treatment and Deepfreeze Chilling

3.3.2 Contour Changes (Warpage) (Cont.)

It is believed the moderate warpage that occurred in the deepfreeze-chilled specimen was due to the more gradual rate of heat loss from the material compared with the rapid loss that occurred during the oil quench.

In conclusion, the results of this evaluation not only indicated the unacceptability of rapid quenching as a production process, but also verified the validity of the currently accepted production procedure of slow uniform cooling subsequent to hot forming operations.

3.3.3 Mechanical Property Changes

Table VIII presents the tabulated mechanical property change data for the ambient air-cooled, the oil-quenched, and the deepfreeze-chilled "cooling rate" specimens. The results of the initial tests of the "mechanically routed" specimens, including the "control specimens," were not only lower than anticipated, but included moderate "data scatter" as well. Critical examination of the specimens with the aid of a 50X microscope revealed extensive "grooving" of the edges and severe pitting of the surfaces of the test sections. The low mechanical properties attained during the initial tensile tests, therefore, were believed due to the combination of these surface conditions and the possible presence of slight surface twinning which had not been completely removed during the etching operation.

The consistently higher results exhibited by the "EDM" specimens appear to substantiate this belief. Not only were the results substantially higher in all cases, but the data were more consistent, with little "data scatter." In addition, the tensile properties of the ambient air-cooled specimens approached those

TABLE VIII

MECHANICAL PROPERTY CHANGES - COOLING RATE SPECIMENS

Specimen			Thickness	Width	Fty		Ftu		Elong.
Type Cooling	Ident.	Prep.	Inch	Inch	Load	PSI	Load	PSI	%
Amb. Air	1-C	Router	.0605	.2462	870	58,408	1055	70,828	4.0
@75°F	1-1	Router	.0597	.2457	835	56,926	890	60,675	2.5
	1-2	Router	.0605	.2485	880	58,533	955	63,521	3.0
	1-3	Router	.0610	.2484	895	59,066	1290	84,868	16.0
Average (2)						58,175		69,688	7.2
	1-4	EDM	.0627	.2428	900	59,119	1290	84,737	10.0
	1-5	EDM	.0624	.2458	910	59,330	1160	75,629	7.0
	1-6	EDM	.0627	.2467	925	59,800	1150	74,347	5.0
Average (2)						59,416		78,238	7.3
Oil Quench	2-C	Router	.0642	.2450	810	51,497	985	62,623	2.5
@88°F	2-1	Router	.0641	.2462	735	46,574	1040	65,900	6.0
	2-2	Router	.0641	.2486	750	47,065	1030	64,636	4.0 (1)
	2-3	Router	.0635	.2487	740	46,858	935	59,205	3.5
Average (2)						46,832		63,247	4.5
	2-4	EDM	.0657	.2482	787	48,262	1130	69,296	6.0
	2-5	EDM	.0652	.2477	790	48,916	1140	70,588	7.0
	2-6	EDM	.0654	.2500	800	48,929	1090	66,666	5.0
Average (2)						48,702		68,850	6.0

(1) Broke outside of the gage length

(2) Control specimens not included

TABLE VIII (Cont.)

MECHANICAL PROPERTY CHANGES - COOLING RATE SPECIMENS

Specimen			Thickness	Width	Fty		Ftu		Elong.
Type Cooling	Ident.	Prep.	Inch	Inch	Load	PSI	Load	PSI	%
Deepfreeze	3-C	Router	.0638	.2486	800	50,439	990	62,418	4.0
@-85°F	3-1	Router	.0638	.2483	742	46,839	995	62,810	4.0
	3-2	Router	.0633	.2467	740	47,387	1030	65,957	6.0
	3-3	Router	.0627	.2474	732	47,189	990	63,822	5.0
Average (2)						47,138		64,196	5.0
	3-4	EDM	.0647	.2462	775	48,653	1080	67,800	5.0
	3-5	EDM	.0637	.2542	800	49,405	1135	70,094	7.0
	3-6	EDM	.0636	.2467	788	50,223	1125	71,701	8.0
Average (2)						49,427		69,865	6.7

(2) Control specimens not included

3.3.3 Mechanical Property Changes (Cont.)

quoted in the vendor certifications. In addition, examination of these specimens with the aid of the same 50X microscope revealed the uniformly superior condition of the surface of the test sections.

The evaluation of the tabulated data indicated that rapid rates of cooling do adversely affect the mechanical properties of beryllium and that carefully controlled uniform slow cooling is required if deleterious effects are to be avoided.

No investigation was made of the possible effect of cooling rate on the bend ductility of the material. It is believed reasonable to assume that some effect would occur. However, the extensive metallographic analyses required for the verification of this assumption are not considered to be within the scope of this program.

SECTION 4.0
ANNEALING

At this time, annealing, per se, is not an accepted beryllium manufacturing fabrication operation. However, the effect of prolonged exposure at elevated temperatures on the basic mechanical properties of beryllium sheet material may be cause for concern when extensive hot-forming operations are contemplated. It is known that prolonged exposure to temperatures in excess of 1400°F will result in the lowering of the mechanical properties of the material as the result of recrystallization and grain growth. This metallurgical change has been the subject of several studies and will not be repeated here.

4.1 INTRODUCTION

The object of this phase of the program was the correlation of prolonged elevated temperature exposure with respect to the mechanical properties of beryllium sheet material. The effects of annealing the material at 1350°F, the nominal forming temperature, and at 1150°F for periods of time up to 250 hours were investigated.

Table IX presents the certified chemical analyses and mechanical properties of the two lots of material procured for this investigation from the Brush Beryllium Company and The Beryllium Corporation in accordance with the requirements of Lockheed Specification LAC 07-4008.

4.2 EXPERIMENTAL PROCEDURE

The test specimens were cut from the sheet material in the longitudinal direction, as determined from the dimensions of the original sheet, i.e., the 4.0-inch dimension of the specimens was parallel to the long side of the parent sheet. The .750" x 4.00" specimens were cut to size, using the abrasive wheel technique, and the loading pin holes were precision drilled in the ends. These holes were used to index the

TABLE IX

CHEMICAL ANALYSES AND MECHANICAL PROPERTIES - ANNEALING SPECIMENS

VENDOR DATA

Material Identification

Vendor	Brush Beryllium Co.	The Beryllium Crop.
Lot. No.	1942	397D
Sheet No.	540A	HR 31-2
Gage	.060	.060

Chemical Analyses - %

Be Assay	98.50	98.22
Be O	1.81	1.91
Fe	.130	.134
Si	.040	.050
Al	.090	.083
Mg	.010	.027
Mn	.010	(1)
Ni	.020	(1)
Cr	.010	(1)
C	.010	.102

Mechanical Properties

Sheet No.	Test Direction	Ftu Psi	Fty Psi	Elong. % in 1"
540A	L	83,000	56,700	9.8
540A	T	84,700	59,800	12.3
HR 31-2	L	70,600	51,200	8.0
HR 31-2	T	78,400	53,000	21.0

(1) .04 maximum

4.2 EXPERIMENTAL PROCEDURE (Cont.)

blank specimens during the EDM (Electrical Discharge Machining) finish machining of the .250" x 1.000" gage sections. Figure 21 illustrates the drill jig, the electrode forming tool with electrodes, and the electrode holder with several completed tensile specimens.

Each edge was cut independently in two stages; i.e., it was cut half way through the sheet from each side. The use of this two-stage procedure not only resulted in reduced electrode wear but, more importantly, in minimum taper of the edges of the gage sections.

The EDM method was selected in preference to the conventional precision machining method for two reasons:

a. Economy -- this method is less costly than precision machining methods, and less susceptible to spalling and surface microcracks.

b. Integrity -- due to the narrow (.250") gage width, the side pressure of a conventional routing tool must be maintained at the minimum level to avoid possible fracture of the specimen; there is no side pressure during the EDM process and, therefore, no possible compromise of the integrity of the gage section. A previous investigation of the EDM process indicated that the very shallow separation, of approximately .0001", along the basal plane was readily removed by subsequent chemical etching.

The EDM method was slow, and a slight mismatch, or "step," occurred at the intersection of the two cuts. Accurate measurements of the specimen gage width, required for subsequent calculations of tensile strength, therefore, were somewhat difficult to obtain.

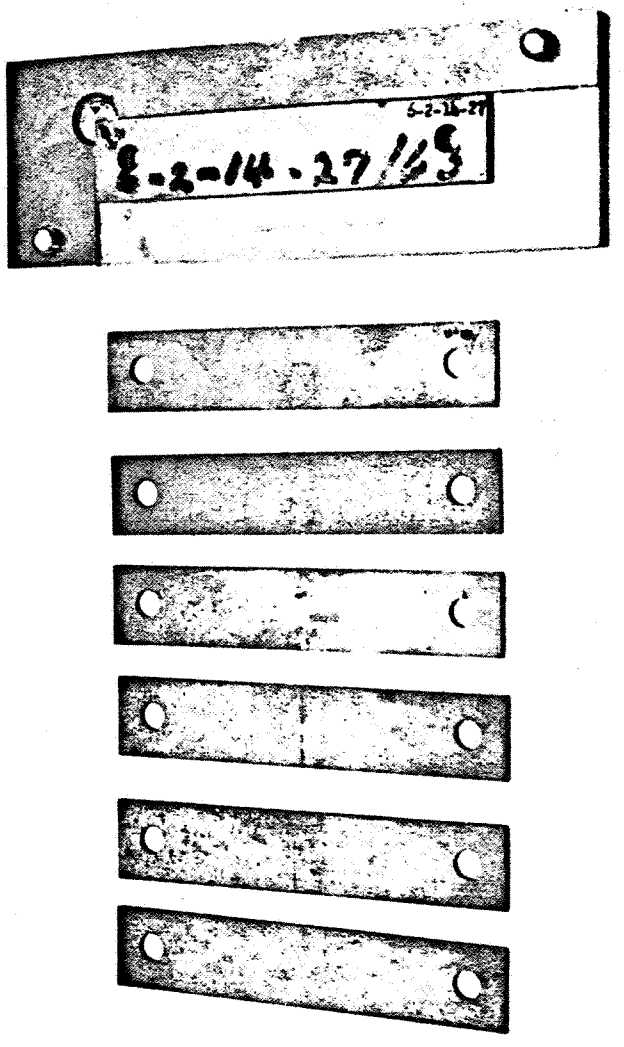
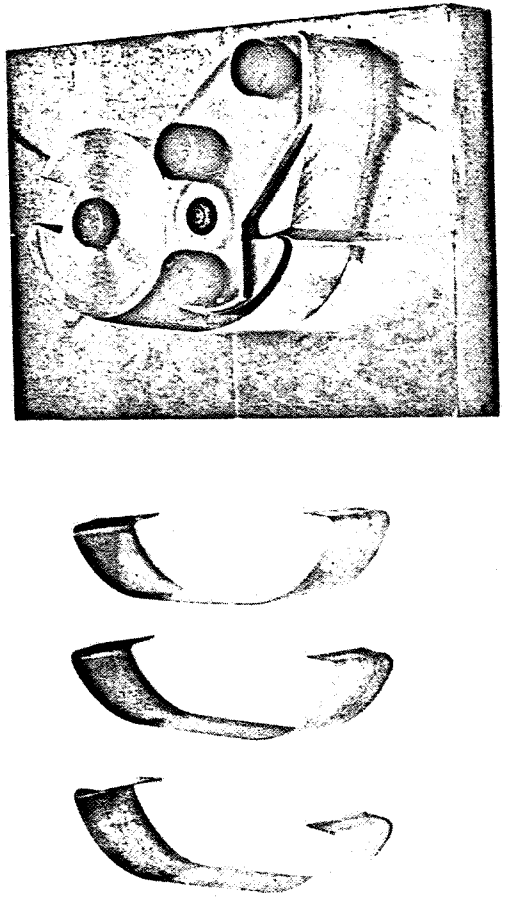
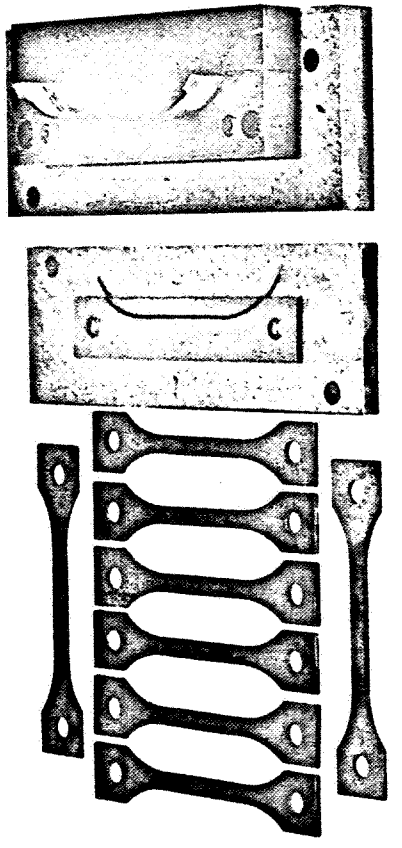


Figure 21. Drill Jig
Drilled Specimens



Electrode Forming Tool
Electrodes



Electrode Holder
Completed Specimens

4.2 EXPERIMENTAL PROCEDURE (Cont.)

The specimens then were annealed in laboratory type furnaces at 1150°F and 1350°F for periods of 2, 4, 8, 16, 50, 100 and 250 hours, and 1/2, 1, 2, 8, 16, 50, 100, and 250 hours, respectively. Three specimens were annealed at each temperature and time increment, followed by light etching to remove the surface oxide film. Apparently the initial etching did not completely remove the heavy oxide film as very small elongations and inconsistent results were obtained during the initial tensile tests. The balance of the specimens then were hand-polished with 240 grit paper and re-etched; approximately .002" of material was removed from each surface to insure the removal of the oxide film and surface imperfections.

All of the tensile tests were performed at room temperature on a 20,000-pound capacity Wiedemann-Baldwin universal testing machine. A Model T-2M Baldwin Microformer non-averaging snap extensometer (1-inch gage length) was used to measure the strain in the specimens; the strain rate, of approximately .005 inch/inch/minute, was controlled by a Baldwin Strain Pacer. The charts of the autographically recorded loads were used to determine the load at 0.2% offset and at failure. The total elongation was determined by fitting the fractured specimens together and measuring the distance between the gage marks.

4.3 RESULTS AND DISCUSSION

Tables X and XI present the tabulated tensile test data; Figures 22 and 23 present the plotted results. All of the data for each temperature, at each increment of time, were plotted individually and average curves were drawn to indicate the general trends. A logarithmic scale was used for the "Time" abscissa to provide a convenient means for including all of the data.

TABLE X

PROLONGED ANNEALING AT 1150°F

Time Hours	Tensile Strength - KSI		Elongation % in 1 inch
	Ultimate	Yield	
0 (Control)	73.7	53.8	10.0
	77.4	57.9	5.4
2	85.1	58.3	21.0
	72.4	52.2	9.0
	83.7	57.8	22.0
4	75.8	54.1	13.0
	74.9	53.1	13.0
	74.1	53.5	10.0
8	76.0	54.0	15.0
	87.6	60.3	20.8
	81.5	60.6	7.0
16	85.9	60.1	19.0
	85.6	60.1	16.8
50	85.7	60.8	14.3
	82.2	61.1	8.0
	71.0	53.7	6.0
100	79.6	60.5	6.0
	85.9	61.1	15.3
	85.0	60.9	13.0
250	85.4	59.5	15.0
	83.8	59.7	12.0
	73.3	60.2	4.0

TABLE XI

PROLONGED ANNEALING AT 1350°F

Time Hours	Tensile Strength - KSI		Elongation % in 1 inch
	Ultimate	Yield	
1/2	73.4	51.4	9.5
	74.4	51.2	15.0
	74.1	50.8	15.0
1	88.1	60.1	23.0
	74.4	52.6	12.5
	79.8	60.7	6.2
2	79.9	59.1	6.2
	81.7	59.9	7.0
	73.4	51.5	12.2
8	72.8	49.2	12.9
	69.4	50.6	7.0
	73.9	49.9	13.0
16	84.9	57.9	14.0
	84.3	56.9	16.7
	86.8	58.2	20.0
50	56.6	45.0	3.0
	69.2	45.2	9.5
	71.6	46.4	12.7
100	71.0	48.0	11.7
	73.1	48.5	15.0
	73.5	48.6	16.0
250	69.7	48.9	8.5
	74.8	49.7	20.0
	72.2	48.5	12.7

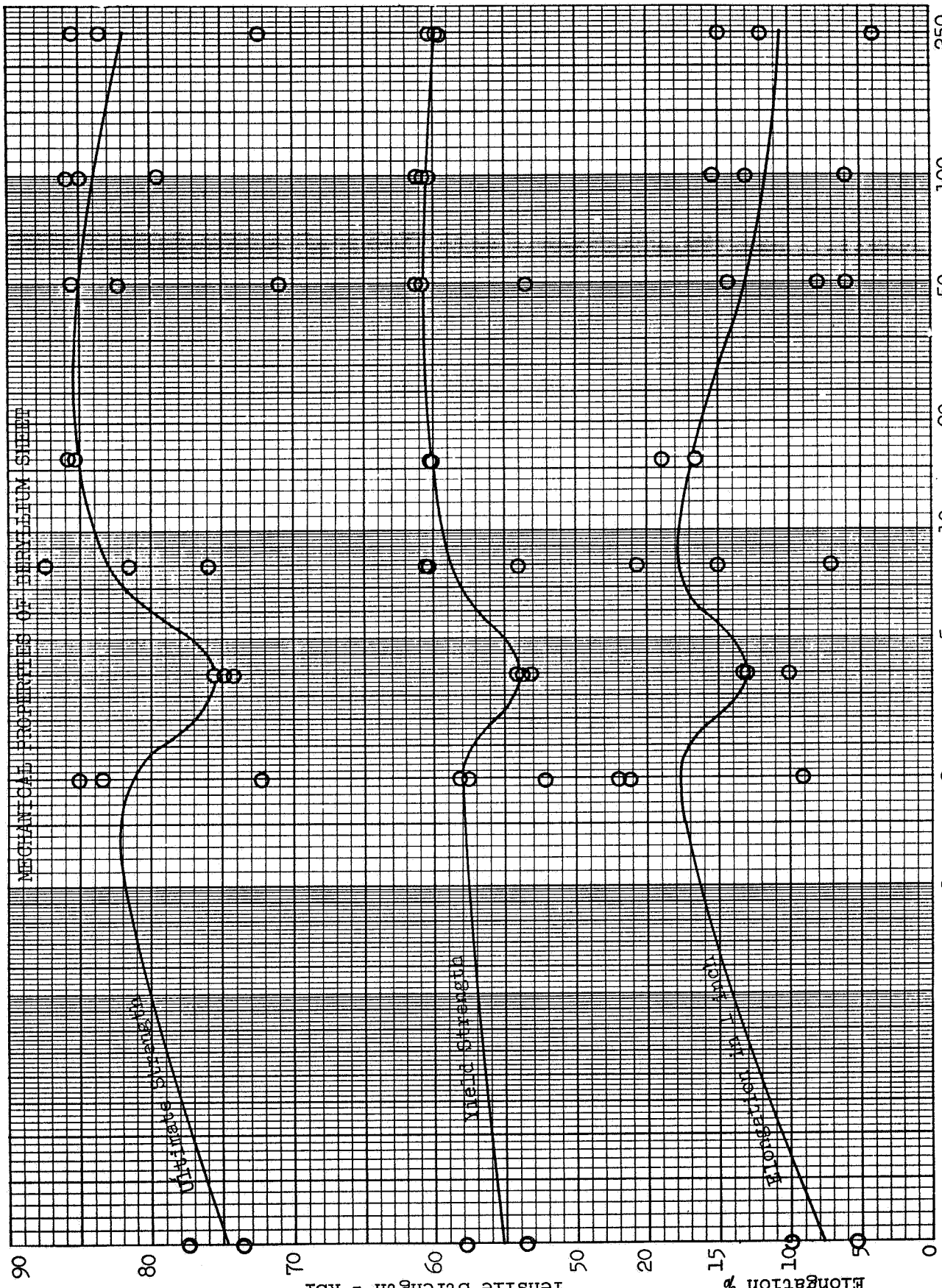


Figure 22. Effect of Prolonged Annealing at 1150°F

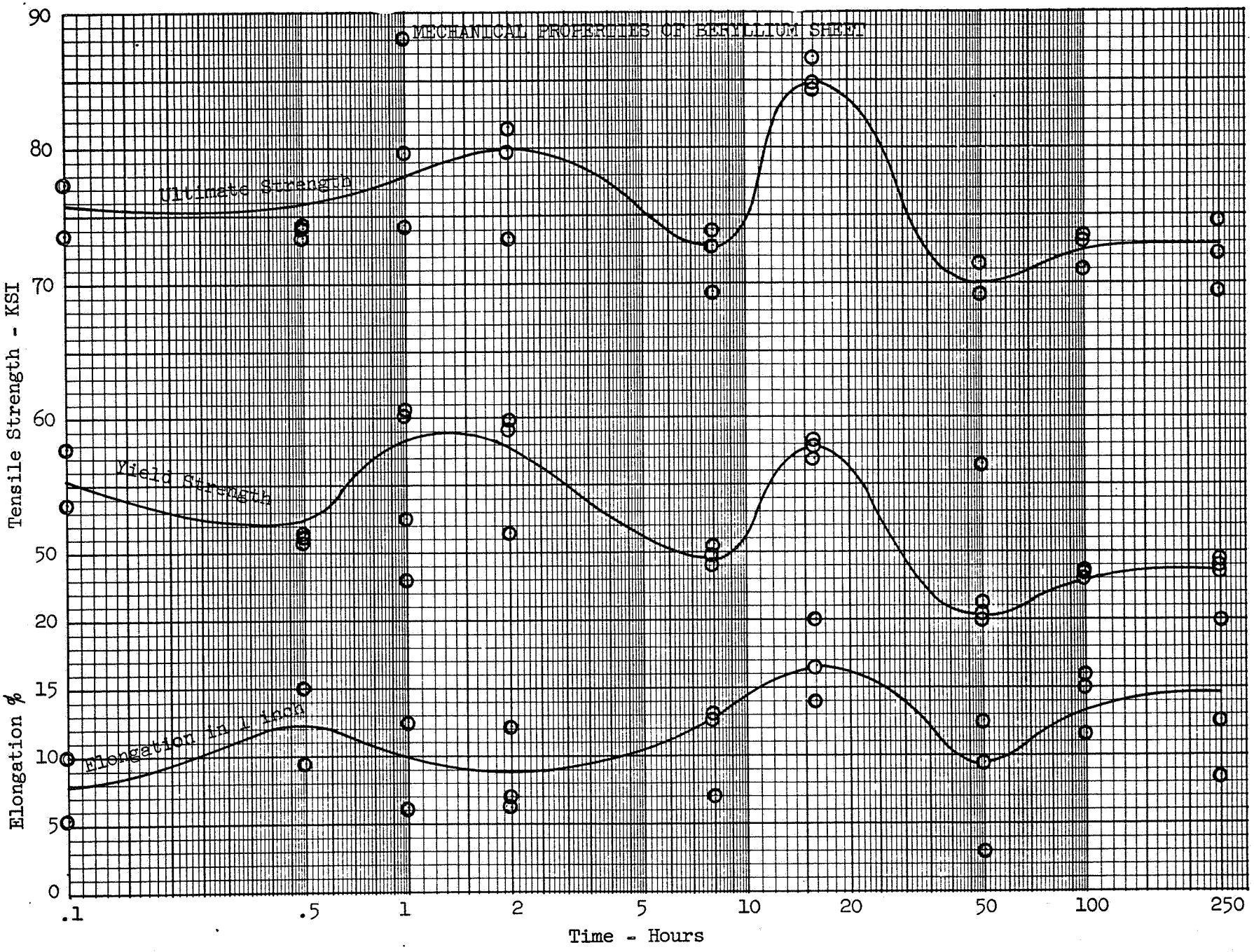


Figure 23. Effect of Prolonged Annealing at 1350°F

4.3 RESULTS AND DISCUSSION (Cont.)

Figures 22 and 23 illustrate the effects of long-time annealing at temperatures of 1150°F and 1350°F, respectively. Although considerable data scatter may be noted, slight, but definite, trends are discernible in the yield and ultimate tensile strengths and in the elongation.

With the exception of the "drop" at 4 hours, the yield and ultimate strengths of the specimens annealed at 1150°F increased slightly with time; reaching the maximum values after approximately 100 hours, and then gradually decreasing to a level still above the initial "as received" levels. The maximum elongation was attained after approximately 10 hours; the "drop" at 4 hours also occurred in the elongation as well as in the strength levels. After 250 hours at the 1150°F temperature, the material properties appear to be approaching the original values. These trends are illustrated in Figure 22. The "4-hour drop," believed due to a metallurgical change, is not considered to be critical as the material properties are still in the "as received" range. The research investigation required for the resolution of this interesting phenomenon is not considered to be within the scope of this program.

As illustrated in Figure 23, the material properties of the specimens annealed at 1350°F displayed somewhat different trends. The yield and ultimate tensile strengths decreased slowly and steadily after an initial peak at approximately 2 hours; the elongation apparently increased to an initial peak at approximately 16 hours, followed by a significant decrease at 50 hours and then a secondary increase. The strength levels also reached peaks at approximately 16 hours; however the general trend, as stated earlier, was definitely downward with time; the strengths after 250 hours were less than "as received" although the elongation was slightly higher. The several maxima and minima displayed by this data are believed due to metallurgical changes; the research effort required for the resolution of these interesting phenomena is not within the scope of this program.

4.3 RESULTS AND DISCUSSION (Cont.)

The wide scatter in the data, at each increment of time, is believed to be due either to the slightly "stepped" edge condition previously discussed, or to the presence of surface micro-cracks that were not completely removed during the final etching operation.

In conclusion, the results of this phase of the program indicate that exposure to normal elevated temperatures for reasonable periods of time; e.g., the conditions to which the material is subjected during normal forming operations, results in negligible deleterious effects on the properties of the material, and that exposures as long as 250 hours at these temperatures do not result in serious degradation of the material properties. However, thermal treatment, or annealing, at 1150°F for approximately 8 hours results in more consistent and predictable effects on the material than a similar treatment at 1350°F. Annealing, per se, does not appear to be a required production process.

SECTION 5.0
SUMMARY

The results of this investigation clearly indicate the extreme importance of the proper preparation of beryllium tensile specimens. The effect of minor imperfections is particularly acute on the smaller gage selections, due to the very small cross-sectional area of the test section.

The results of these brief investigations indicate the following conclusions:

1. The relative thermal coefficients of expansion of beryllium and of stress relief holding fixtures must be given careful consideration during the design phase of the tools.

2. A more effective thermal treatment for the relief of residual stresses appears to be 1025°F for approximately 4 hours rather than the commonly accepted short treatment conditions of 1350°F for approximately 20-30 minutes.

3. Except for very large or extremely precise parts, the dimensional changes due to rapid cooling are negligible.

4. Rapid cooling has a deleterious effect on both the contour and the mechanical properties of beryllium sheet material.

5. Rapid cooling, or quenching, is not an acceptable beryllium production process.

6. The validity of the currently accepted production procedure of slow uniform cooling subsequent to hot forming operations has been verified. This procedure results in minimum warpage and minimum deleterious effects on the mechanical properties of beryllium.

SECTION 5.0 (Cont.)

7. The exposure of beryllium to normal, elevated temperatures for a reasonable period of time results in negligible deleterious effects on the properties of the material.

8. Thermal treatment, or annealing, at 1150°F for approximately 8 hours appears to result in more consistent and predictable effects than similar treatment at 1350°F.

9. Annealing, per se, does not appear to be a required production process.